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Frequency of Leak Occurence for Fittings in Synthetic Organic Chemical Plant Process Units

Radian Corp.
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16. ABSTRACT The report summarizes results of a study of the frequency of leak occurrence in the synthetic chemical manufacturing industry (SOCMI). The frequency of occurrence of fugitive emissions was determined by instrumental screening, defined as measurement of the concentration of volatile organic compounds (VOCs) as close as possible to the point of suspected leakage (e.g., the valve stem, pump seal). The screening value is the maximum repeatable VOC concentration detected for any given source expressed in parts per million by volume (ppmv). A total of 24 process units in the SOCMI were tested. The frequency of fugitive emissions sources with screening values equal to or greater than 10,000 ppmv was tabulated for all units combined, for each type of chemical process, and for each process unit.

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Processing	Stationary Sources	13H
Leakage	Fugitive Emissions	14G
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FREQUENCY OF LEAK OCCURRENCE FOR FITTINGS IN SYNTHETIC ORGANIC CHEMICAL PLANT PROCESS UNITS

September 1980

by

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CONTENTS

Section		Page
Figures		iv
Tables		v
1.	INTRODUCTION	1
	1.1 Technical Objectives and Approach	2
	1.2 Process Units Screened	2
2.	SUMMARY OF RESULTS	4
3.	TEST PLAN	8
4.	METHODOLOGY	11
	4.1 Execution	11
	4.2 Quality Control Checks	13
	4.2.1 Instrument Calibration	13
	4.2.2 Repeat Screening	15
	4.2.3 Replicate Screening	15
	4.3 Screening Problems	18
	4.3.1 Source Accessibility	18
	4.3.2 Instrument Problems	21
	4.3.3 Other Problems	24
5.	DETAILED RESULTS	25
6.	REFERENCES	40
APPENDICES		
A - Adipic Acid -- Units 35 and 64		A-1
B - Acetaldehyde -- Unit 33		B-1
C - Acetone/Phenol -- Unit 12		C-1

CONTENTS (continued)

Section	Page
D - Acrylonitrile -- Units 65 and 66	D-1
E - Chlorinated Ethanes -- Units 60, 61, and 62	E-1
F - Cumene -- Units 5 and 6	F-1
G - Ethylene -- Units 11, 4, and 2	G-1
H - Methyl Ethyl Ketone -- Units 31 and 32	H-1
I - Formaldehyde -- Unit 22	I-1
J - Methyl Methacrylate -- Unit 34	J-1
K - Vinyl Acetate -- Units 1 and 3	K-1
L - Vinyl Chloride Monomer/Ethylene Dichloride -- Units 28, 29, 20, and 21	L-1

FIGURES

Number		Page
4-1	Screening data sheet	14
4-2	Repeat screening trend - Example A	16
4-3	Repeat screening trend - Example B	17
4-4	Comparison of Radian replicate screening values with contractor screening values	19

TABLES

Number		Page
1-1	Chemical Process Units Screened for Fugitive Emissions.....	3
2-1	Summary of SOCMI Process Unit Fugitive Emissions.....	5
2-2	Average Source Screening Rates.....	7
3-1	Factors Considered in Selection of Types of Process Units.....	9
4-1	Summary Replicate Screening Values During Quality Control Checks.....	20
5-1	Fugitive Emission Profile with Confidence Intervals - Adipic Acid.....	27
5-2	Fugitive Emission Profile with Confidence Intervals - Acetaldehyde.....	28
5-3	Fugitive Emission Profile with Confidence Intervals - Acetone/Phenol.....	29
5-4	Fugitive Emission Profile with Confidence Intervals - Acrylonitrile.....	30
5-5	Fugitive Emission Profile with Confidence Intervals - Chlorinated Ethanes.....	31
5-6	Fugitive Emission Profile with Confidence Intervals - Cumene.....	32
5-7	Fugitive Emission Profile with Confidence Intervals - Ethylene.....	33
5-8	Fugitive Emission Profile with Confidence Intervals - Methyl Ethyl Ketone.....	34
5-9	Fugitive Emission Profile with Confidence Intervals - Formaldehyde.....	35

TABLES (continued)

Number		Page
5-10	Fugitive Emission Profile with Confidence Intervals - Methyl Methacrylate	36
5-11	Fugitive Emission Profile with Confidence Intervals - Vinyl Acetate	37
5-12	Fugitive Emission Profile with Confidence Intervals - Vinyl Chloride Monomer	38
5-13	Fugitive Emission Profile with Confidence Intervals - Ethylene Dichloride	39

SECTION 1

INTRODUCTION

The U. S. EPA Industrial Environmental Research Laboratory, Research Triangle Park, NC, has coordinated a study to develop information about fugitive emissions, *i.e.*, unintentional discharges or leaks, of volatile organic compounds from equipment sources in the synthetic organic chemical manufacturing industry (SOCMI). The results of this study will be used by the EPA in the evaluation of regulatory strategies for the control of fugitive emissions from SOCMI process units (the SOCMI is defined as those process units which produce, as intermediate or final products, any of the chemicals listed in the draft Background Information Document (1) for fugitive emission control standards). Fugitive emission sources studied include process valves, pump seals, compressor seals, flanges, relief valves, open-ended lines, process drains, and agitator seals.

This report presents findings of the fugitive emission study. The technical objective of this program, the approach used in fulfilling this objective and the process units studied in this program are reviewed in Section 1. The results of the program are summarized in Section 2. The test plan, or the rationale used in the selection of the process units to be studied, is detailed in Section 3. Procedures followed and problems encountered during acquisition of fugitive emission data are discussed in Section 4. The test results are presented in Section 5. The Appendices present descriptions of the various processes, discussions of sampling problems, data on sampling time, and the results of the fugitive emission study on a unit-by-unit basis.

1.1 TECHNICAL OBJECTIVES AND APPROACH

The objective of the program was to develop data on the frequency of occurrence of fugitive emissions from various sources in the SOCMI. This objective was met by a two-fold approach:

- the acquisition of fugitive emission data by the four participating contractors (EPA Contract Number):
  --Acurex Corporation (68-02-3176-01, 06),
  --PEDCo Environmental, Inc. (68-02-3173-02, 11),
  --Radian Corporation (68-02-3171-01), and
  --TRW, Inc., Energy Systems Group (68-02-3174-04); and
- coordination of the program, administration of quality control checks, data processing, and preparation of the final report by Radian Corporation.

For this program, "screening" refers either to the measurement of fugitive emissions at a single source or to the overall sampling of a process unit. "Screening value" refers to the maximum repeatable concentration of total hydrocarbons detected at a source with a portable hydrocarbon detector, expressed in parts per million by volume (ppmv). "Screening data" refers to the information recorded for each screened source, including the type of source, the screening value, line temperature and pressure, ambient conditions, etc.

1.2 PROCESS UNITS SCREENED

The frequency of occurrence of component leaks was investigated for 14 types of chemical process units. A total of 24 process units were screened by the four contractors. The screening took place over a six-month period (1/22/80 - 8/1/80); an average of about one week was spent in each process unit. The rationale used in the selection of these process units is discussed in Section 3. Table 1-1 presents the process unit identification (ID) number, the type of process, and the contractor for each of the 24 process units.

TABLE 1-1. CHEMICAL PROCESS UNITS SCREENED FOR FUGITIVE EMISSIONS

Unit No.	Unit Type	Contractor
1	Vinyl Acetate	Radian
2	Ethylene	Radian
3	Vinyl Acetate	Radian
4	Ethylene	Radian
5	Cumene	Radian
6	Cumene	Radian
11	Ethylene	TRW
12	Acetone/Phenol	TRW
20	Ethylene Dichloride	PEDCo
21	Vinyl Chloride Monomer	PEDCo
22	Formaldehyde	PEDCo
28	Ethylene Dichloride	PEDCo
29	Vinyl Chloride Monomer	PEDCo
31	Methyl Ethyl Ketone	Acurex
32	Methyl Ethyl Ketone	Acurex
33	Acetaldehyde	Acurex
34	Methyl Methacrylate	Acurex
35	Adipic Acid	Acurex
60	Trichloroethylene/Perchloroethylene	PEDCo
61	1,1,1-Trichloroethane	PEDCo
62	Ethylene Dichloride	PEDCo
64	Adipic Acid	PEDCo
65	Acrylonitrile	PEDCo
66	Acrylonitrile	PEDCo

SECTION 2

SUMMARY OF RESULTS

Results of the fugitive emission screening program conducted at the 24 process units are summarized in Table 2-1. The following information is shown for the total population of each source type:

- the number of sources screened (Column 1),
- the percentage of the total identified sources that were not screened (Column 2),
- the percentage of screened sources with screening values greater than or equal to 10,000 ppmv (Column 3), and
- the 95 percent confidence interval for the percentage of screened sources with screening values greater than or equal to 10,000 ppmv (Column 4).

The 95 percent confidence interval, denoted by the upper and lower limits in Column 4, is the interval of percentage values that can be said, with 95 percent certainty, to include the true percentage value.

The sources are further categorized by phase of the material contained by the source. For this program, "gas" refers to gas phase at process conditions. Liquids were classified based on their most volatile component present at 20 weight percent or more. If that component had a vapor pressure equal to or greater than 0.04 psi (0.3 kPa) at 20°C, the material was classified as a "light liquid"; if not, the material was classified as a "heavy liquid" (1 kPa = 0.145 psi = 7.50 mmHg).

TABLE 2-1. SUMMARY OF SOCMI PROCESS UNIT FUGITIVE EMISSIONS

Source Type	Service	(1) Number Screened	(2) % Not Screened	(3) % of Screened Sources with Screening Values ≥ 10,000 ppmv	(4) 95% Confidence Interval for Percentage of Sources ≥ 10,000 ppmv
Flanges	Gas	1,443	4.6	4.6	(3.6, 5.8)
	Light Liquid	2,897	2.6	1.2	(0.9, 1.8)
	Heavy Liquid	607	2.4	0.0	(0.0, 0.6)
Process Drains	Gas	83	23.1	2.4	(0.3, 8.4)
	Light Liquid	527	1.9	3.8	(2.3, 5.8)
	Heavy Liquid	28	0.0	7.1	(0.9, 23.5)
Open Ended Lines	Gas	923	17.5	5.8	(4.4, 7.5)
	Light Liquid	3,603	[illegible].4	3.9	(3.3, 4.6)
	Heavy Liquid	477	21.5	1.3	(0.5, 2.8)
Agitator Seals	Gas	7	46.1	14.3	(0.4, 57.9)
	Light Liquid	8	11.1	0.0	(0.0, 36.9)
	Heavy Liquid	1	66.7	0.0	(0.0, 97.5)
Relief Valves	Gas	85	72.7	3.5	(0.7, 10.0)
	Light Liquid	69	40.5	2.9	(0.3, 10.1)
	Heavy Liquid	3	66.7	0.0	(0.0, 70.8)
Valves	Gas	9,668	17.5	11.4	(10.8, 12.1)
	Light Liquid	18,294	12.2	6.4	(6.1, 6.8)
	Heavy Liquid	3,632	9.9	0.4	(0.2, 0.7)
Pumps	Light Liquid	647	4.3	8.8	(6.6, 11.1)
	Heavy Liquid	97	40.5	2.1	(0.3, 7.3)
Compressors	Gas	29	9.4	6.9	(0.9, 22.8)
Other[1]	Gas	19	9.5	21.0	(6.0, 45.6)
	Light Liquid	33	19.5	6.1	(0.7, 20.2)
	Heavy Liquid	2	33.3	0.0	(0.0, 84.2)

[1]Includes filters, vacuum breakers, expansion joints, rupture disks, sight glass seals, etc.

The sources in this study were screened at an average rate of 1.7 minutes per source for a two-person team (or 3.4 person-minutes per source). This average screening rate includes time spent for instrument calibration and repair. Table 2-2 provides data on screening time on a unit-by-unit basis.

TABLE 2-2. AVERAGE SOURCE SCREENING RATES

Process Unit	Number of Screened Sources	Average Screening Time Per Source, Minutes[1]
1	1,391	2.0
2	5,078	1.3
3	2,780	0.9
4	5,278	1.5
5	1,025	0.9
6	1,573	1.0
11	3,685	1.9
12	3,207	3.2
20	1,430	2.6
21	868	
22	230	1.8
28	744	1.6
29	2,619	
31	585	1.6
32	679	2.2
33	1,148	1.2
34	2,019	0.9
35	1,577	0.7
60	2,720	1.6
61	570	
62	42	
64	664	1.9
65	1,406	2.5
66	1,864	1.9
TOTAL	43,182	1.7

[1]Average source screening time was determined for a two-person team, one person screening with a portable hydrocarbon detector and one person recording data. Average screening time includes time spent for instrument calibration, maintenance, and repair.

SECTION 3

TEST PLAN

The process units studied were selected to represent a cross section of the population of the SOCMI. Several factors were considered during process unit selection. These factors included total annual production volume, number of producers, volatility, toxicity, and value of the final chemical product. Factors varied widely from unit type to unit type, so that the selected process unit types represented a reasonable sample of the variety of chemical process units encountered in the SOCMI.

A major factor was the total annual production volume by all domestic manufacturers of the chemical product under consideration. Chemical process units were selected so that the total production volumes of the individual chemical products ranged widely. Although minor specialty chemicals with very low production volumes were not included, the process unit types that were selected had production volumes that varied substantially--from trichloroethylene's 0.3×10^9 lb/year to ethylene's 25.4×10^9 lb/year in 1977. The variation in total production volumes of the chemicals produced in the selected process units is shown in Table 3-1.

Table 3-1 also shows that the chemicals produced in the selected process unit types were manufactured by varying numbers of producers. The number of producers of a given chemical does not necessarily reflect the total number of process units, as a producer may operate more than one process unit. The number of producers ranged from three manufacturers of methyl methacrylate to 31 manufacturers of ethylene.

TABLE 3-1. FACTORS CONSIDERED IN SELECTION OF TYPES OF PROCESS UNITS

Chemical product	Production volume, 10^9 lb/yr (1977)[1]	Number of producers (1977)[1]	Vapor pressure of product, mm Hg at 20°C[2]	Threshold limit value[5] of product, ppmv[3]	Cost of product, $/lb (1980)[4]
Acetaldehyde	≤1.7	5	760+	100	0.29
Acetone	2.2	13	160.5	1000	0.30
Acrylonitrile	1.6	4	84.9	20(skin)	0.38
Adipic acid	1.5	5	<1.1	NA[6]	0.53
Cumene	2.6	14	3.3	50(skin)	0.27
Ethylene	25.4	31	760+	NA[6]	0.24
Ethylene dichloride	11.0	12	60.2	10	0.14
Formaldehyde	6.0	16	760+	2	0.07
Methyl ethyl ketone	0.5	5	76.2	200	0.35
Methyl methacrylate	0.7	3	28.1	100	0.50
Perchloroethylene	0.6	8	13.7	100(skin)	0.23
Phenol	2.3	13	<1.7	5(skin)	0.36
1,1,1-Trichloroethane	0.6	3	95.6	350	0.31
Trichloroethylene	0.3	5	57.0	100	0.27
Vinyl acetate	1.6	6	84.2	10	0.34
Vinyl chloride monomer	6.0	12	760+	5	0.22

[1]Reference (2)

[2]Reference (3)

[3]Reference (4)

[4]Reference (5)

[5]Eight-hour time weighted average

[6]Threshold limit value not available or not established

A third factor considered in the selection of the process units was volatility of the chemicals being processed. Volatilities of chemical products at 20°C, expressed as vapor pressure in Table 3-1, ranged from less than 1.1 mm Hg (0.15 kPa) for adipic acid to 760^{+} mm Hg (100^{+} kPa) for gaseous ethylene.

Toxicity of the chemicals being processed was also a consideration during selection of the process units. Threshold limit values (TLVs) ranged from 2 ppmv for formaldehyde to 1,000 ppmv for acetone (eight-hour weighted averages). The TLVs for the chemicals produced by the selected process units are shown in Table 3-1.

Cost of the chemical product was another factor considered in the selection of the process unit types. Process units were selected that produced chemicals of widely differing values, ranging from $0.07/lb of formaldehyde to $0.53/lb of adipic acid (June 1980 prices), as shown in Table 3-1.

Variations in other factors, such as line pressures, line temperatures, and age of the process units, were observed during study of the selected process units. Stream pressures ranged from vacuum service to above 1,500 psig. Process temperatures ranged from below -250°F to over 1,500°F. The ages of the selected process units ranged from less than two years to greater than 15 years.

SECTION 4

METHODOLOGY

The procedures followed and problems encountered during acquisition of the fugitive emission screening data are presented in Section 4.

4.1 EXECUTION

The screening procedures began with the definition of the process unit boundaries. All feed streams, reaction/separation facilities, and product and by-product delivery lines were identified on process flow diagrams and in the process unit. Process data, including stream composition, line temperature, and line pressure, were obtained for all flow streams.

Each process stream was then traced in the field. In most cases, plant personnel assisted the screening team in obtaining process data and identifying sources to be screened. Accessible sources were screened by a two-person team (one person handling the hydrocarbon detector and one person recording data) as the sources were encountered along the stream. Tags or markings were placed on screened sources to prevent inadvertent rescreening.

The Century Systems Models OVA-108 and OVA-128 hydrocarbon detectors were used for screening. The HNU Systems, Inc., Model PI 101 Photoionization Analyzer was also used to screen sources at the formaldehyde process unit. The detector probe of the instrument was placed directly on those areas of the sources where leakage would typically occur. For example, gate valves were screened along the circumference of the annular area around the valve stem where the stem exits the packing gland and at the packing gland/valve bonnet interface.

The maximum concentration reading (in ppmv) on the instrument was recorded for each source. For some sources, the reading fluctuated considerably. In these cases, the maximum reading repeated at least once was recorded as the screening value.

All accessible sources of the following source types were screened:

- process valves,
- pump seals,
- compressor seals,
- agitator seals,
- relief valves,
- process drains, and
- open-ended lines.

Also, a randomly selected subset of flanges was screened. Originally, only five percent of all flanges were screened. The subset was increased to 20 percent of all flanges when initial results indicated a higher frequency of measurable emissions than had been encountered in previous programs. For the purposes of this program, "flange" referred to any pipe-to-pipe or tubing-to-tubing connection, excluding welded joints.

The following data were recorded for each identified source:

- source ID number;
- source type;
- screening value (ppmv) if the source was accessible;
- type of service (gas, light liquid, or heavy liquid);
- composition of material in the line (identities and mole fractions of the two major organic components);
- line temperature (°F);
- line pressure (psig);

- ambient air temperature (°F); and
- source elevation (number of landings above ground level).

A copy of the screening data sheet used in the project is shown as Figure 4-1.

Only those sources containing at least one weight percent VOC were screened or identified as inaccessible. Fittings in steam and cooling water service were thus eliminated from consideration. Wastewater and process water fittings were screened if they contained at least one weight percent VOC.

4.2 QUALITY CONTROL CHECKS

A three-part quality control strategy was used to ensure experimental consistency within the acquired fugitive emission data base. These three procedures were as follow:

- daily instrument calibrations,
- determinations of screening repeatability, and
- replicate screening by Radian quality control personnel.

4.2.1 Instrument Calibration

Each screening instrument was calibrated on a daily basis, at minimum. The Model OVA-108 instruments, with a logarithmic scale reading from 1 ppmv to 10,000 ppmv, were calibrated with high (8,000 ppmv) and low (500 ppmv) concentration methane-in-air standards to ensure accurate operation at both ends of the instrument's range. The Model OVA-128 instruments, with a linear readout ranging from 0 ppmv to 1,000 ppmv, were also calibrated with high and low concentration standards. A pre-calibrated dilution probe was required with the OVA-128 when calibrating with the 8,000 ppmv standard.

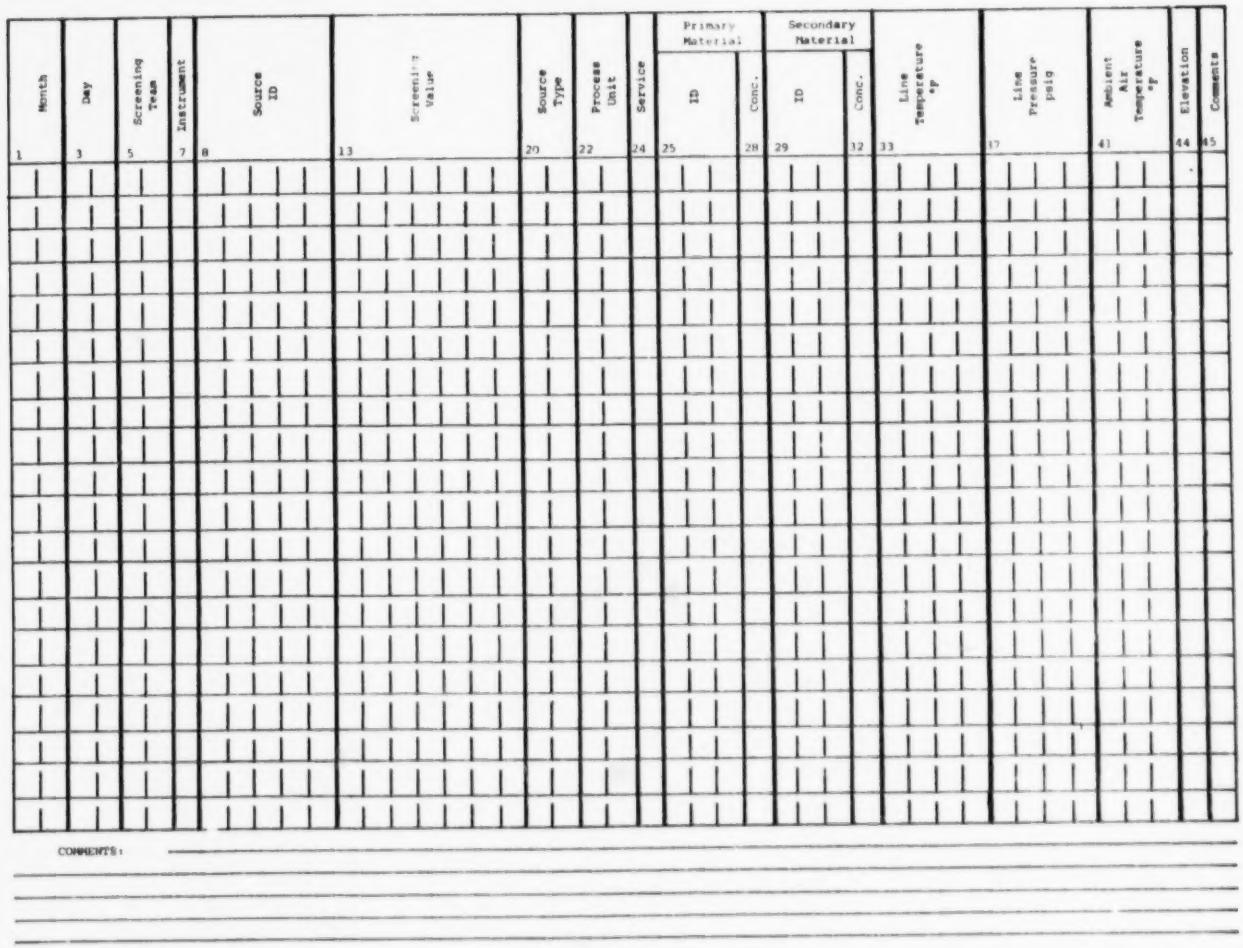

Figure 4-1. Screening data sheet.

The HNU Photoionization instrument, used to screen the formaldehyde process unit, was calibrated with isobutylene, which has an ionization potential close to that of formaldehyde. The linear scale of the HNU ranges from 0 to 2,000 ppmv. Dilution probes, used to extend the range of the Models OVA-108 and OVA-128, were calibrated at a 10:1 ratio, so that screening values up to 100,000 ppmv and 10,000 ppmv, respectively, could be obtained.

Results of the calibration of the instruments with the high and low concentration standards and of the dilution probe were recorded each day.

4.2.2 Repeat Screening

Three valves were selected on the first day of screening at each process unit to be studied for screening repeatability. One of the three selected valves had an initial screening value between 200 ppmv and 1,000 ppmv, another between 1,000 ppmv and 10,000 ppmv, and the third above 10,000 ppmv. Each selected valve was screened three times each day, within a 30-minute period. The three screening values for each of the three valves were recorded each day. Examples of two different repeat screening trends are shown in Figures 4-2 and 4-3.

Variance component analysis of the repeat screening data was performed. This analysis indicated that the between-day variation in screening value exceeds the within-day (actually within-30 minute) variation by only 9.2 percent for a given valve. Statistical analysis of the repeat screening data also showed that ±85 percent is the maximum difference to be expected (90 percent of the time) between two screening values obtained for a given valve within 30 minutes.

4.2.3 Replicate Screening

Replicate screening procedures were carried out by Radian Corporation to determine if differences in instrument operation, calibration techniques,

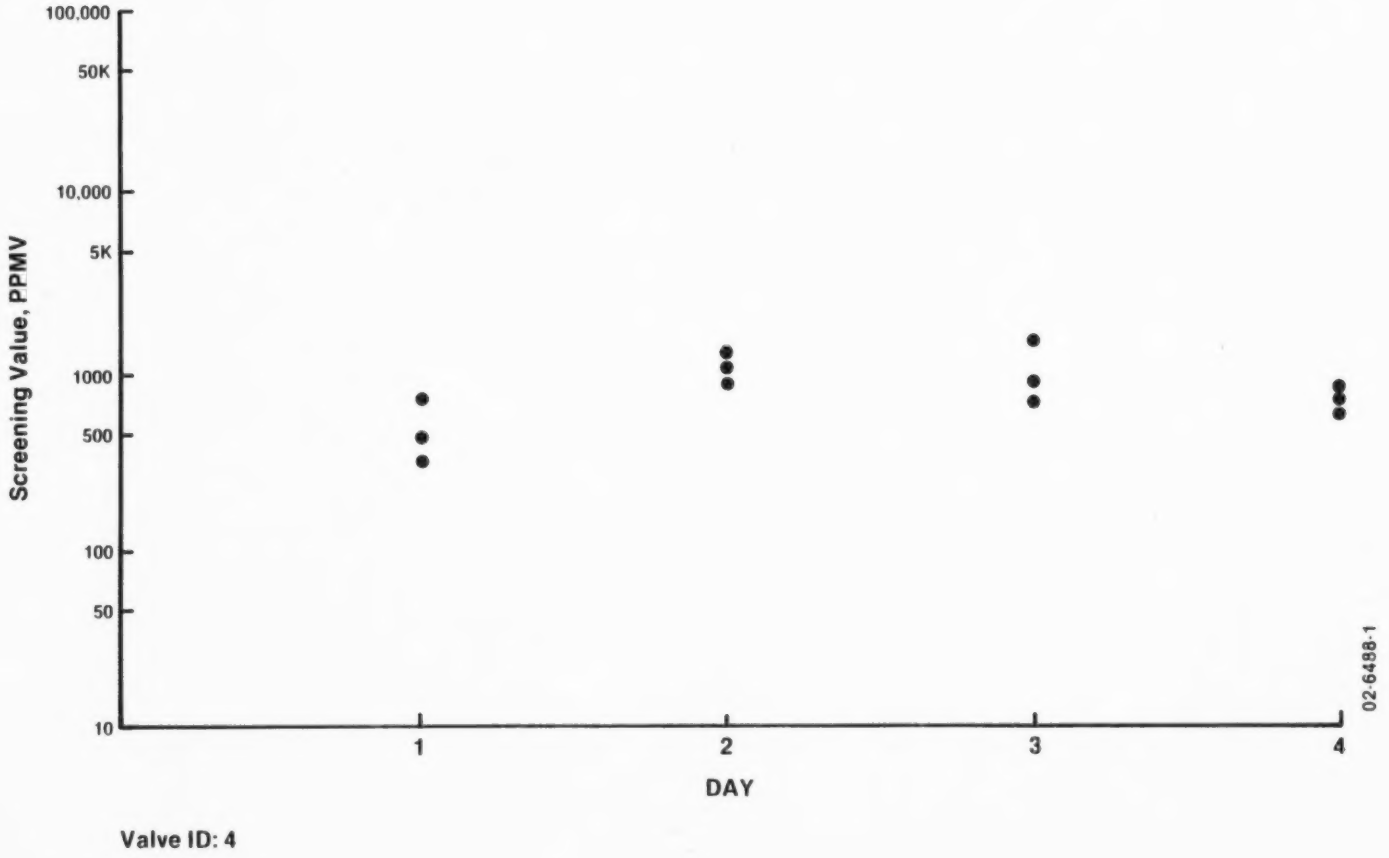

Figure 4-2. Repeat screening trend - Example A

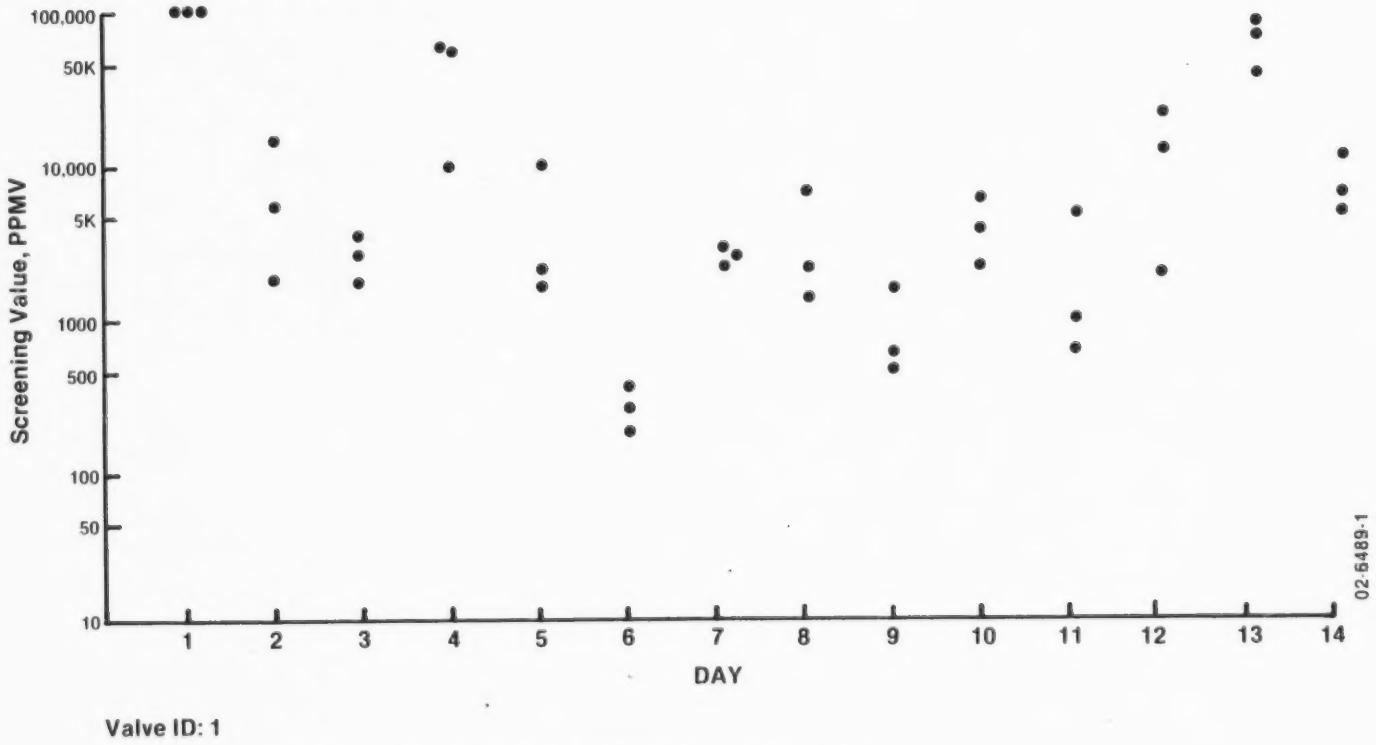

Figure 4-3. Repeat screening trend - Example B

screening techniques, and data recording techniques existed among the four contractors. These quality control checks were done only at the first process unit entered by each contractor.

No major discrepancies in instrument operation or procedural techniques were noted. Also, agreement between screening values recorded by Radian and by the other contractors was good, as shown in Figure 4-4. Results of the replicate screenings are summarized in Table 4-1.

4.3 SCREENING PROBLEMS

Certain problems or circumstances prevented screening access to various sources or delayed the overall screening effort. Discussions of the most important of these problems, source accessibility and instrument reliability, are presented in the following subsections.

4.3.1 Source Accessibility

In-plant safety regulations were rigorously observed and movable ladders were generally not used, so approximately 13 percent of the sources in VOC service were not screened because of their location.

For example, certain valves and open-ended lines were in elevated locations that could only be reached with a ladder. Other valves and open-ended lines could only have been accessed with major effort, such as the use of extensive scaffolding, a crane, or a "cherry picker".

Heavy ice formations on many valves in cold service prevented or hampered attempts to obtain screening values. Fugitive emissions from these valves were often difficult to detect, as the leak often surfaced at random locations around the ice cap covering the valve. Also, accurate screening values for these ice-covered valves were difficult to obtain; care had to be taken to prevent moisture from being drawn from wet surfaces into the instrument probe.

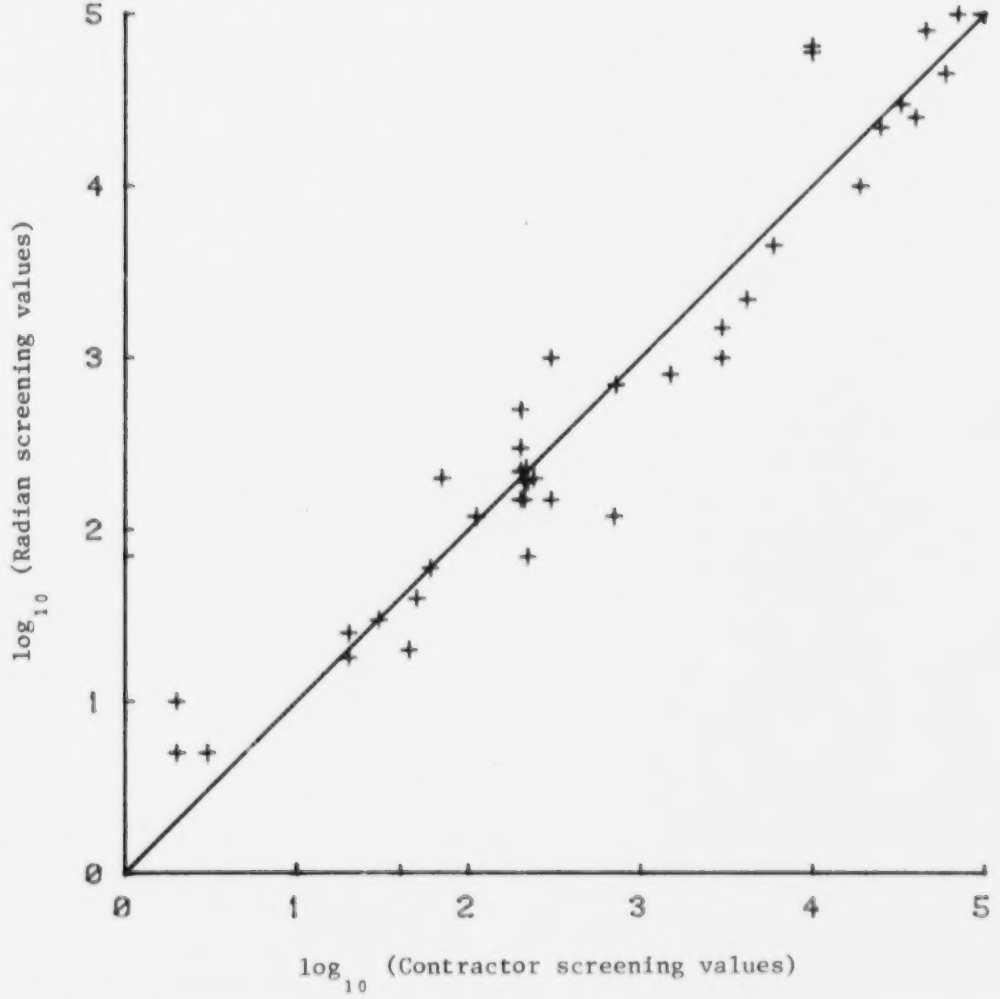

Figure 4-4. Comparison of Radian replicate screening values with contractor screening values.

TABLE 4-1. SUMMARY REPLICATE SCREENING VALUES DURING QUALITY CONTROL CHECKS

Source ID	Contractor	Contractor screening value (ppmv)	Radian screening value (ppmv)	Comments
35	PEDCo(Team 20)	720;1100;1200	700	QC Sources; Radian screening ≈2 hours after PEDCo
35	PEDCo(Team 23)	720;600;700	700	
1292	PEDCo	1500;1800;1800	800	
1317	PEDCo	60	60	
384	PEDCo	210	190	
389	PEDCo	4200	2200	
379	PEDCo	0	70	Screened ≈1 hour after PEDCo. 35 other sources were screened at zero by both contractors.
-	PEDCo	0	0	
29	Acurex	10K;25K;19K	65K;22K;10K	QC Source
30	Acurex	300;110;700	150;120;120	QC Source
50	Acurex	210;220;220	150;70;190	QC Source
120	Acurex	300	1000	
121	Acurex	3000	1000	
124	Acurex	6000	4500	
126	Acurex	70	200	
145	Acurex	200	300	
147	Acurex	3000	1500	
-	Acurex	0	0	13 other sources were screened at zero by both contractors
17	TRW	10K;40K;46K	60K;25K;80K	QC Source
34	TRW	200;240;216	220;200;230	QC Source
76	TRW	100K;70K;98K	100K;100K;100K	QC Source
578	TRW	45	20	
579	TRW	3	5	
580	TRW	2	5	
583	TRW	60,000	45,000	
584	TRW	10	0	
585	TRW	33,000	30,000	
605	TRW	2	10	
606	TRW	40	0	
608	TRW	50	40	
609	TRW	30	30	
610	TRW	20	25	
611	TRW	20	18	
618	TRW	200	150	
619	TRW	200	500	
-	TRW	0	0	22 other sources were screened at zero by both contractors

Certain locations of valves and open-ended lines were unsafe for screening. These locations included areas heavily obstructed by hot piping, areas in the path of escaping process steam, and other areas of the process deemed unsafe by plant safety rules for personnel access (*e.g.*, areas adjacent to the oxygen injection equipment at a vinyl acetate process unit).

Most pressure relief valves vented directly to a flare header and were not screened. Many of the relief valves which did vent to the atmosphere could not be accessed without a major effort.

The seal areas of about 60 percent of the compressors were shrouded and vented either back to the process or to a flare header. Because the seal areas were not accessible, these compressors were either not screened or were screened at the interface between the compressor impeller/blade housing and the shroud enclosing the seal. The seal areas of 20 percent of the compressors were open to the atmosphere but were covered by a safety guard. These seal areas were screened with the hydrocarbon detector probe placed as close as possible to the seal area.

Safety guards also prevented direct access to approximately 20 percent of the pump seals. These pump seals were screened with the detector probe held as close as possible to the seal areas.

4.3.2 Instrument Problems

The screening effort at several process units was delayed by two categories of instrument problems--major part failure and "nuisance" malfunctions.

Parts or service from the factory or factory representatives were required for failure of major parts such as the battery pack, preamplifier, and readout meter. Problems due to major part failure were aggravated by slow instrument service by the factory. During the screening program, the

Century Systems factory facilities were being moved to a location in another state, causing significant delays in delivery of ordered parts and repair of defective instruments. Timely response from regional factory representatives and access to back-up instruments prevented many significant delays because of major part failure. The service delays experienced when major parts failed led to identification of a list of critical spare instrument parts. These parts include the readout meter, battery pack, regulator repair kit, pressure gauges, hydrogen flow valves, and filters.

Nuisance malfunctions were often corrected on site by repair or replacement of defective parts. An example of this type of problem was the repair of instrument hydrogen leaks. Certain nuisance malfunctions were tolerated until more extensive repair could be made, providing the malfunction only delayed the screening effort and did not compromise the quality of the data. Erratic operation of the ignitor plug was an example of this type of malfunction.

Another example of a nuisance malfunction occurred when excessive amounts of moisture or organic liquids were drawn into the probe of the Century Systems hydrocarbon detectors. Water drawn into the instrument probe and internals during rainy weather or from icy surfaces often produced random electrical signals and subsequent erratic readout behavior. The problem was corrected by allowing the instrument to run and dry out for several hours before reuse. Organic liquids drawn into the instrument saturated the sample lines and caused hydrogen flame-out. Flushing of the instrument's sample lines was necessary before re-ignition of the hydrogen flame was possible.

The Century Systems instruments, which operate on the flame ionization detection (FID) principle, contain an internal hydrogen flame for combustion and ionization of VOC's that are drawn into the detector probe. Ignition of the hydrogen flame on certain instruments was difficult. Inspection of the ignitor plug on these instruments indicated erratic and weak "glowing" of the

resistance heating element. Ignition of the hydrogen flame was necessary not only during instrument start-up but also after the flame was extinguished because of introduction of a "too-rich", *i.e.*, very high, concentration of VOC's (typically greater than 100,000 ppmv).

Faulty hydrogen flow regulation on certain instruments resulted in variable hydrogen flow rate to the combustion chamber and subsequent drift in readout accuracy. Frequent instrument calibration (as often as hourly on one occasion) was necessary to eliminate substantial readout error. Also, certain instruments experienced random calibration drift, even when no variance in hydrogen flow rate was observed.

The Century Systems instruments displayed sluggish response to certain chemicals, such as benzene, cumene, and phenol. When the instrument probe was introduced to a concentration of these chemicals at a leaking source, an abnormally long period of time (30 to 60 seconds) elapsed before the instrument readout stabilized at a maximum concentration. Also, the instrument's recovery from the maximum concentration to the ambient, or background, concentration was very slow (one to three minutes). The instruments registered no response at all to other chemicals, such as adipic acid.

Short lengths of Tygon tubing were attached to the instrument probes and the dilution probes to prevent the tips of the probes from being fouled. The Tygon tubing apparently adsorbed certain hydrocarbons when the probes were exposed to high concentrations of these hydrocarbons, biasing the response of the instrument. The problem was resolved by replacing the Tygon tubing with Teflon tubing. The Tygon tubing may also have been partly responsible for the sluggish response of the instruments to certain chemicals. Chemicals adsorbed on the tubing may have gradually evaporated and been drawn into the instrument, causing a slow recovery to ambient concentrations.

Manipulation of the Model OVA-128 decade multiplier switch, particularly with gloved hands, was awkward and time-consuming. For this reason, the OVA-128 was a less efficient screening instrument than the OVA-108, which displayed concentrations up to full scale without manipulation of instrument controls. Also, a dilution probe calibrated at 100:1 dilution, which is beyond the design range of the probe, was required to extend the range of the Model OVA-128 from 1,000 ppmv to 100,000 ppmv. However, for presenting results with 10,000 ppmv as the critical maximum concentration as in Table 2-1, no significant problems arise from using a 100:1 dilution with the OVA-128.

4.3.3 Other Problems

High background concentrations of VOC's in some areas of certain process units delayed the screening effort or prevented the obtaining of screening data. At the least, high backgrounds masked concentrations from leaking sources or prevented the accurate measurement of comparatively low concentrations. At worst, high background concentrations caused discomfort to screening personnel or compromised their safety.

Adverse weather conditions, *i.e.*, heavy rain, high wind, or severe cold, hampered the screening effort at several process units. In some cases, weather conditions only slowed the screening effort. In other cases, however, wet weather conditions resulted in instrument failure or the inability to record data legibly, thus halting screening completely.

SECTION 5

DETAILED RESULTS

The detailed results of the screening program are presented in two types of tables. These tables are referred to as Fugitive Emission Profile tables, presented in this section, and Summary Statistics tables, which are found in the appendices.

An example of a Fugitive Emission Profile table is Table 5-1, which presents data for adipic acid process units 35 and 64. Three data entries, comprising a "cell", are presented for various source/service combinations. The first cell in Table 5-1 is 0.0%/(0.0, 7.2)/49. The first data entry in the cell, 0.0%, represents the combined percentage of flanges in gas service in units 35 and 64 that had measured screening values of greater than or equal to 10,000 ppmv. The second data entry in the cell, (0.0, 7.2), is the 95 percent confidence interval. This means that it can be stated with 95 percent certainty that the *true* percentage of sources with screening values greater than or equal to 10,000 ppmv in this population of screened flanges lies within the interval of 0.0 percent and 7.2 percent. The uncertainty in the estimated percentage of sources with screening values greater than or equal to 10,000 ppmv, reflected by the confidence interval, stems from random fluctuations in source emission rates, instrument operation, weather conditions, and other factors. The third data entry in the cell, 49, is the number of flanges in gas service screened at units 35 and 64. As indicated in Table 5-1, the confidence interval narrows with increasing number of screened sources.

The Data Summary sections of Appendix A provide Summary Statistics tables for the two adipic acid units (process units 35 and 64). These tables

provide additional information concerning the percentages of sources with screening values greater than or equal to 1,000 ppmv and numbers of sources not screened because of source inaccessibility and other factors.

Table 5-2 through Table 5-13 present Fugitive Emission Profile tables for the remaining process units. The Summary Statistics table for each process unit is found in the appropriate appendix.

Data for process unit 62, an ethylene dichloride unit, are grouped in Table 5-5 with miscellaneous chlorinated ethanes data, rather than with ethylene dichloride unit data in Table 5-13. This grouping was made because only a small portion of unit 62 was screened. Therefore, screening data for unit 62 do not characterize an entire ethylene dichloride unit, as do screening data for units 20 and 28 in Table 5-13.

As stated above, the appendices contain the Summary Statistics for each unit screened. In addition, the appendices provide information on:

- process description,
- sampling problems and observations, and
- sampling time.

The appendices were prepared by the contractor responsible for screening the specific unit (see Table 1-1).

TABLE 5-1. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - ADIPIC ACID

Source	Service	Weighted Average	Unit #35	Unit #64
Flanges	Gas	0.0%/(0.0, 7.2)/49	0.0%/(0.0, 21.8)/15	0.0%/(0.0, 10.3)/34
	Light Liquid	0.0%/(0.0, 84.2)/2	0.0%/(0.0, 84.2)/2	-
	Heavy Liquid	0.0%/(0.0, 1.2)/320	0.0%/(0.0, 1.7)/218	0.0%/(0.0, 3.6)/102
Process Drains	Heavy Liquid	0.0%/(0.0, 30.8)/10	0.0%/(0.0, 30.8)/10	-
Open Ended Lines	Gas	0.0%/(0.0, 17.6)/19	0.0%/(0.0, 97.5)/1	0.0%/(0.0, 18.5)/18
	Light Liquid	0.0%/(0.0, 97.5)/1	0.0%/(0.0, 97.5)/1	-
	Heavy Liquid	0.0%/(0.0, 1.8)/214	0.0%/(0.0, 3.6)/102	0.0%/(0.0, 3.2)/112
Relief Valves	Gas	0.0%/(0.0, 84.2)/2	0.0%/(0.0, 84.2)/2	-
Valves	Gas	0.0%/(0.0, 3.8)/95	0.0%/(0.0, 7.9)/45	0.0%/(0.0, 7.1)/50
	Light Liquid	0.0%/(0.0, 19.5)/17	0.0%/(0.0, 19.5)/17	-
	Heavy Liquid	0.0%/(0.0, 0.3)/1478	0.0%/(0.0, 0.3)/1163	0.0%/(0.0, 1.2)/315
Pumps	Heavy Liquid	0.0%/(0.0, 11.6)/30	0.0%/(0.0, 97.5)/1	0.0%/(0.0, 11.9)/29
Compressors	Gas	0.0%/(0.0, 97.5)/1	-	0.0%/(0.0, 97.5)/1
Other[1]	Gas	0.0%/(0.0, 84.2)/2	-	0.0%/(0.0, 84.2)/2
	Heavy Liquid	0.0%/(0.0, 97.5)/1	-	0.0%/(0.0, 97.5)/1

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

[1]Includes rotary drum seal and tank hatch.

TABLE 5-2. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - ACETALDEHYDE

Source	Service	Unit #33
Flanges	Gas	0.0%/(0.0, 10.9)/32
	Light Liquid	0.0%/(0.0, 2.5)/144
Process Drains	Light Liquid	41.7%/(15.2, 72.3)/12
Open Ended Lines	Gas	8.8%/(1.9, 23.7)/34
	Light Liquid	5.1%/(2.2, 9.8)/158
Relief Valves	Gas	0.0%/(0.0, 60.2)/4
	Light Liquid	0.0%/(0.0, 70.8)/3
Valves	Gas	4.5%/(1.9, 8.7)/178
	Light Liquid	0.5%/(0.1, 1.6)/551
Pumps	Light Liquid	9.4%/(2.0, 25.0)/32

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

TABLE 5-3. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - ACETONE/PHENOL

Source	Service	Unit #12
Flanges	Light Liquid	0.0%/(0.0, 4.4)/82
	Heavy Liquid	0.0%/(0.0, 11.6)/30
Process Drains	Light Liquid	20.0%/(2.5, 55.6)/10
Open Ended Lines	Gas	0.0%/(0.0, 84.2)/2
	Light Liquid	1.5%/(0.7, 3.0)/518
	Heavy Liquid	0.0%/(0.0, 3.4)/107
Relief Valves	Light Liquid	0.0%/(0.0, 17.7)/19
	Heavy Liquid	0.0%/(0.0, 70.8)/3
Valves	Gas	0.0%/(0.0, 36.9)/8
	Light Liquid	0.3%/(0.1, 0.7)/1818
	Heavy Liquid	0.0%/(0.0, 0.8)/488
Pumps	Light Liquid	2.3%/(0.3, 8.2)/86
	Heavy Liquid	0.0%/(0.0, 9.7)/36

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

TABLE 5-4. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - ACRYLONITRILE

Source	Service	Weighted Average	Unit #65	Unit #66
Flanges	Gas	1.4%/(0.2, 5.0)/142	1.3%/(0.0, 7.2)/75	1.5%/(0.0, 8.0)/67
	Light Liquid	0.0%/(0.0, 1.0)/382	0.0%/(0.0, 3.1)/117	0.0%/(0.0, 1.4)/265
	Heavy Liquid	0.0%/(0.0, 12.3)/28	0.0%/(0.0, 12.3)/28	-
Process Drains	Light Liquid	12.5%/(0.3, 52.7)/8	100.0%/(2.5, 100.0)/1	0.0%/(0.0, 41.0)/7
	Heavy Liquid	100.0%/(2.5, 100.0)/1	100.0%/(2.5, 100.0)/1	-
Open Ended Lines	Gas	0.9%/(0.0, 4.7)/116	0.0%/(0.0, 6.3)/57	1.7%/(0.0, 9.1)/59
	Light Liquid	2.5%/(1.3, 4.3)/486	3.7%/(1.3, 8.0)/160	1.8%/(0.7, 4.1)/326
	Heavy Liquid	10.5%/(2.9, 24.8)/38	10.5%/(2.9, 24.8)/38	-
Valves	Gas	2.4%/(1.0, 4.3)/396	4.4%/(2.0, 8.3)/203	0.0%/(0.0, 1.9)/193
	Light Liquid	1.9%/(1.2, 2.7)/1494	2.5%/(1.4, 4.1)/591	1.4%/(0.8, 2.4)/903
	Heavy Liquid	0.0%/(0.0, 3.8)/95	0.0%/(0.0, 3.8)/95	-
Pumps	Light Liquid	8.2%/(2.7, 18.1)/61	5.3%/(0.1, 26.0)/19	9.5%/(2.7, 22.6)/42
	Heavy Liquid	25.0%/(3.2, 65.1)/8	25.0%/(3.2, 65.1)/8	-
Compressors	Gas	0.0%/(0.0, 84.2)/2	0.0%/(0.0, 84.2)/2	-
Other[1]	Gas	12.5%/(0.3, 52.7)/8	12.5%/(0.3, 52.7)/8	-
	Light Liquid	0.0%/(0.0, 60.2)/4	0.0%/(0.0, 84.2)/2	0.0%/(0.0, 84.2)/2
	Heavy Liquid	0.0%/(0.0, 97.5)/1	0.0%/(0.0, 97.5)/1	-

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

[1]Includes rupture disk, head gasket, mixer, and atmospheric vent.

TABLE 5-5. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - CHLORINATED ETHANES

Source	Service	Unit #60 Trichloroethylene/ Perchloroethylene	Unit #61 1,1,1-Trichloroethane	Unit #62 Ethylene Dichloride
Flanges	Gas	0.0%/(0.0, 28.5)/11	-	0.0%/(0.0, 52.2)/5
	Light Liquid	0.0%/(0.0, 0.7)/525	0.0%/(0.0, 4.9)/73	-
	Heavy Liquid	0.0%/(0.0, 84.2)/2	-	-
Process Drains	Light Liquid	0.0%/(0.0, 33.6)/9	-	-
Open Ended Lines	Gas	0.0%/(0.0, 30.8)/10	-	0.0%/(0.0, 19.5)/17
	Light Liquid	1.5%/(0.5, 3.2)/412	1.8%/(0.2, 6.4)/111	-
	Heavy Liquid	0.0%/(0.0, 60.2)/4	-	-
Agitator Seals	Light Liquid	-	0.0%/(0.0, 97.5)/1	-
Relief Valves	Light Liquid	0.0%/(0.0, 70.8)/3	-	-
Valves	Gas	0.0%/(0.0, 12.3)/28	-	0.0%/(0.0, 16.8)/20
	Light Liquid	0.1%/(0.0, 0.3)/1620	1.1%/(0.3, 2.8)/373	-
	Heavy Liquid	0.0%/(0.0, 26.5)/12	-	-
Pumps	Light Liquid	8.3%/(2.8, 18.4)/60	10.0%/(0.2, 44.5)/10	-
Other[1]	Gas	33.3%/(0.0, 90.6)/3	-	-
	Light Liquid	9.5%/(1.2, 30.4)/21	0.0%/(0.0, 84.2)/2	-

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

[1]Includes expansion joint, rupture disk, and sight glass seal.

TABLE 5-6. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - CUMENE

Source	Service	Weighted Average	Unit #5	Unit #6
Flanges	Gas	5.2%/(3.2, 8.1)/367	1.0%/(0.0, 5.5)/100	6.7%/(4.1, 10.5)/267
	Light Liquid	1.6%/(0.7, 3.0)/568	0.3%/(0.0, 1.8)/317	3.2%/(1.4, 6.2)/251
	Heavy Liquid	0.0%/(0.0, 2.8)/130	0.0%/(0.0, 6.2)/58	0.0%/(0.0, 5.0)/72
Process Drains	Gas	0.0%/(0.0, 45.9)/6	-	0.0%/(0.0, 45.9)/6
	Light Liquid	3.2%/(0.1, 16.7)/31	3.2%/(0.1, 16.7)/31	-
Open Ended Lines	Gas	0.0%/(0.0, 45.9)/6	0.0%/(0.0, 84.2)/2	0.0%/(0.0, 60.2)/4
	Light Liquid	13.3%/(1.7, 40.5)/15	0.0%/(0.0, 45.9)/6	22.2%/(2.8, 60.0)/9
	Heavy Liquid	0.0%/(0.0, 97.5)/1	-	0.0%/(0.0, 97.5)/1
Relief Valves	Gas	100%/(2.5, 100)/1	100%/(2.5, 100)/1	-
Valves	Gas	14.1%/(11.1, 17.8)/448	4.6%/(1.5, 10.7)/108	17.1%/(13.5, 21.9)/340
	Light Liquid	10.5%/(8.5, 12.8)/799	11.0%/(7.9, 15.1)/328	10.2%/(7.7, 13.4)/471
	Heavy Liquid	0.0%/(0.0, 1.8)/198	0.0%/(0.0, 5.7)/63	0.0%/(0.0, 2.7)/135
Pumps	Light Liquid	16.0%/(4.5, 36.1)/25	30.0%/(6.7, 65.2)/10	6.7%/(0.2, 32.0)/15
	Heavy Liquid	0.0%/(0.0, 70.8)/3	0.0%/(0.0, 97.5)/1	0.0%/(0.0, 84.2)/2

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

TABLE 5-7. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - ETHYLENE

Source	Service	Weighted Average	Unit #2	Unit #4	Unit #11
Flanges	Gas	6.2%/(4.5, 8.5)/627	3.5%/(1.1, 8.1)/142	6.7%/(4.6, 9.5)/448	10.8%/(3.0, 25.4)/37
	Light Liquid	6.1%/(4.0, 8.9)/407	2.0%/(0.1, 10.5)/51	6.9%/(4.5, 10.3)/335	4.8%/(0.1, 23.8)/21
	Heavy Liquid	0.0%/(0.0, 4.1)/89	0.0%/(0.0, 4.9)/73	0.0%/(0.0, 21.8)/15	0.0%/(0.0, 97.5)/1
Process Drains	Gas	12.5%/(0.3, 52.6)/8	50.0%/(1.3, 98.7)/2	-	0.0%/(0.0, 45.9)/6
	Light Liquid	1.0%/(0.3, 2.5)/407	0.0%/(0.0, 3.8)/94	0.0%/(0.0, 1.2)/300	30.8%/(9.1, 61.4)/13
	Heavy Liquid	7.1%/(0.2, 33.9)/14	7.1%/(0.2, 33.9)/14	-	-
Open Ended Lines	Gas	12.1%/(8.7, 16.4)/305	22.0%/(11.5, 36.0)/50	18.3%/(10.1, 29.3)/71	7.1%/(3.8, 11.8)/184
	Light Liquid	19.2%/(11.8, 21.1)/214	31.6%/(17.5, 48.6)/38	16.7%/(7.9, 29.3)/54	16.4%/(10.3, 24.2)/122
	Heavy Liquid	0.0%/(0.0, 4.0)/91	0.0%/(0.0, 4.1)/88	0.0%/(0.0, 70.8)/3	-
Relief Valves	Gas	3.9%/(0.5, 13.5)/51	-	-	3.9%/(0.5, 13.5)/51
	Light Liquid	9.1%/(0.2, 41.3)/11	-	-	9.1%/(0.2, 41.3)/11
Valves	Gas	14.8%/(13.9, 15.8)/6294	14.8%/(13.2, 16.5)/2153	20.1%/(18.1, 22.1)/1970	10.2%/(8.9, 11.6)/2171
	Light Liquid	23.2%/(21.8, 24.7)/4176	25.9%/(23.1, 29.0)/1191	24.2%/(22.1, 26.5)/1990	17.9%/(15.6, 20.4)/995
	Heavy Liquid	1.1%/(0.6, 1.8)/1237	1.0%/(0.5, 1.7)/1144	3.7%/(0.5, 12.8)/54	0.0%/(0.0, 9.0)/39
Pumps	Light Liquid	25.6%/(16.4, 36.8)/78	37.5%/(15.2, 64.6)/16	28.6%/(14.6, 46.3)/35	14.8%/(14.2, 33.7)/27
	Heavy Liquid	0.0%/(0.0, 21.8)/15	0.0%/(0.0, 21.8)/15	-	-
Compressors	Gas	5.9%/(0.2, 28.7)/17	14.3%/(0.4, 57.9)/7	0.0%/(0.0, 70.8)/3	0.0%/(0.0, 41.0)/7

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

TABLE 5-8. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - METHYL ETHYL KETONE

Source	Service	Weighted Average	Unit #31	Unit #32
Flanges	Gas	0.0%/(0.0, 15.4)/22	0.0%/(0.0, 45.9)/6	0.0%/(0.0, 20.6)/16
	Light Liquid	0.0%/(0.0, 4.7)/76	0.0%/(0.0, 10.0)/35	0.0%/(0.0, 8.6)/41
Process Drains	Gas	16.7%/(0.4, 64.1)/6	16.7%/(0.4, 64.1)/6	-
	Light Liquid	21.7%/(7.5, 43.7)/23	21.4%/(4.7, 50.8)/14	22.2%/(2.8, 60.0)/9
Open Ended Lines	Gas	8.1%/(1.7, 21.9)/37	8.3%/(0.2, 38.5)/12	8.0%/(1.0, 26.0)/25
	Light Liquid	10.2%/(6.3, 15.5)/186	8.6%/(3.8, 16.2)/93	11.8%/(6.1, 20.2)/93
Agitator Seals	Gas	0.0%/(0.0, 97.5)/1	0.0%/(0.0, 97.5)/1	-
Relief Valves	Gas	0.0%/(0.0, 70.8)/3	0.0%/(0.0, 97.5)/1	0.0%/(0.0, 84.2)/2
Valves	Gas	9.2%/(5.7, 14.1)/207	18.5%/(10.7, 28.7)/81	3.2%/(0.9, 7.9)/126
	Light Liquid	5.1%/(3.6, 7.1)/671	5.3%/(3.2, 8.5)/321	4.9%/(2.9, 7.8)/350
Pumps	Light Liquid	3.2%/(0.1, 16.7)/31	0.0%/(0.0, 21.8)/15	6.2%/(0.2, 30.2)/16
Compressors	Gas	100%/(2.5, 100)/1	-	100%/(2.5, 100)/1

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

TABLE 5-9. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - FORMALDEHYDE

Source	Service	Unit #22
Flanges	Gas	0.0%/(0.0, 84.2)/2
	Light Liquid	12.5%/(0.3, 52.6)/8
Open Ended Lines	Gas	0.0%/(0.0, 23.2)/14
	Light Liquid	0.0%/(0.0, 9.7)/36
Valves	Gas	2.4%/(0.1, 12.9)/41
	Light Liquid	0.0%/(0.0, 3.0)/121
Pumps	Light Liquid	0.0%/(0.0, 36.9)/8

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

TABLE 5-10. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - METHYL METHACRYLATE

Source	Service	Unit #34
Flanges	Gas	0.0%/(0.0, 9.2)/38
	Light Liquid	0.0%/(0.0, 1.5)/247
Process Drains	Light Liquid	0.0%/(0.0, 16.1)/21
Open Ended Lines	Gas	0.0%/(0.0, 5.7)/63
	Light Liquid	0.3%/(0.0, 1.7)/335
Agitator Seals	Gas	33.3%/(0.0, 90.6)/3
	Light Liquid	0.0%/(0.0, 70.8)/3
Relief Valves	Gas	0.0%/(0.0, 60.2)/4
	Light Liquid	8.3%/(0.2, 38.5)/12
Valves	Gas	0.0%/(0.0, 1.9)/190
	Light Liquid	0.1%/(0.0, 0.5)/1058
Pumps	Light Liquid	4.4%/(0.5, 15.1)/45

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

TABLE 5-11. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - VINYL ACETATE

Source	Service	Weighted Average	Unit #1	Unit #3
Flanges	Gas	2.8%/(0.6, 8.0)/107	2.6%/(0.1, 13.8)/38	2.9%/(0.4, 10.1)/2
	Light Liquid	0.0%/(0.0, 2.1)/173	0.0%/(0.0, 6.4)/56	0.0%/(0.0, 3.2)/117
	Heavy Liquid	0.0%/(0.0, 36.9)/8	0.0%/(0.0, 70.8)/3	0.0%/(0.0, 52.2)/5
Process Drains	Gas	0.0%/(0.0, 5.7)/63	-	0.0%/(0.0, 5.7)/63
	Light Liquid	0.0%/(0.0, 97.5)/1	-	0.0%/(0.0, 97.5)/1
	Heavy Liquid	0.0%/(0.0, 70.8)/3	-	0.0%/(0.0, 70.8)/3
Open Ended Lines	Gas	5.5%/(2.4, 10.6)/145	2.7%/(0.1, 14.2)/37	6.5%/(2.6, 13.0)/108
	Light Liquid	2.5%/(1.1, 5.0)/318	0.0%/(0.0, 7.7)/46	2.9%/(1.3, 5.8)/272
	Heavy Liquid	9.1%/(1.1, 29.2)/22	0.0%/(0.0, 36.9)/8	14.3%/(1.8, 42.8)/14
Agitator Seals	Gas	0.0%/(0.0, 97.5)/1	-	0.0%/(0.0, 97.5)/1
	Light Liquid	0.0%/(0.0, 70.8)/3	0.0%/(0.0, 70.8)/3	-
	Heavy Liquid	0.0%/(0.0, 97.5)/1	0.0%/(0.0, 97.5)/1	-
Relief Valves	Gas	0.0%/(0.0, 97.5)/1	-	0.0%/(0.0, 97.5)/1
	Light Liquid	0.0%/(0.0, 84.2)/2	0.0%/(0.0, 84.2)/2	-
Valves	Gas	3.7%/(2.6, 5.1)/949	3.6%/(1.9, 6.4)/329	3.7%/(2.4, 5.5)/620
	Light Liquid	0.4%/(0.2, 0.7)/2136	0.1%/(0.0, 0.8)/736	0.5%/(0.2, 1.0)/1400
	Heavy Liquid	0.0%/(0.0, 2.9)/124	0.0%/(0.0, 5.4)/67	0.0%/(0.0, 6.3)/57
Pumps	Light Liquid	4.5%/(1.2, 11.1)/89	6.8%/(1.4, 18.7)/44	2.2%/(0.1, 11.8)/45
	Heavy Liquid	0.0%/(0.0, 52.2)/5	0.0%/(0.0, 52.2)/5	-
Compressors	Gas	0.0%/(0.0, 36.9)/8	0.0%/(0.0, 60.2)/4	0.0%/(0.0, 60.2)/4
Other[1]	Gas	16.7%/(0.4, 64.1)/6	16.7%/(0.4, 64.1)6	-
	Light Liquid	0.0%/(0.0, 45.9)/6	0.0%/(0.0, 45.9)/6	-

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

[1]Includes filter and vacuum breaker.

TABLE 5-12. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - VINYL CHLORIDE MONOMER

Source	Service	Weighted Average	Unit #21	Unit #29
Flanges	Gas	4.0%/(0.1, 20.4)/25	0.0%/(0.0, 84.2)/2	4.3%/(0.1, 22.0)/23
	Light Liquid	0.6%/(0.0, 3.4)/163	0.0%/(0.0, 7.9)/45	0.8%/(0.0, 4.6)/118
Open Ended Lines	Gas	0.0%/(0.0, 3.6)/100	0.0%/(0.0, 60.2)/4	0.0%/(0.0, 3.8)/96
	Light Liquid	3.4%/(2.0, 5.5)/473	4.8%/(0.6, 16.2)/42	3.2%/(1.8, 5.5)/431
Relief Valves	Gas	0.0%/(0.0, 41.0)/7	0.0%/(0.0, 45.9)/6	0.0%/(0.0, 97.5)/1
	Light Liquid	0.0%/(0.0, 30.8)/10	0.0%/(0.0, 30.8)/10	-
Valves	Gas	1.0%/(0.3, 2.5)/402	0.0%/(0.0, 4.6)/79	1.2%/(0.3, 3.2)/323
	Light Liquid	1.1%/(0.7, 1.6)/2250	0.0%/(0.0, 0.6)/670	1.5%/(1.0, 2.3)/1580
Pumps	Light Liquid	5.3%/(1.1, 14.6)/57	0.0%/(0.0, 30.8)/10	6.4%/(1.3, 17.5)/47

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

TABLE 5-13. FUGITIVE EMISSION PROFILE WITH CONFIDENCE INTERVALS - ETHYLENE DICHLORIDE

Source	Service	Weighted Average	Unit #20	Unit #28
Flanges	Gas	12.5%/(1.6, 38.4)/16	0.0%/(0.0, 30.8)/10	33.3%/(4.3, 77.7)/6
	Light Liquid	0.0%/(0.0, 7.6)/47	0.0%/(0.0, 8.8)/40	0.0%/(0.0, 41.0)/7
Process Drains	Light Liquid	40.0%/(5.3, 85.3)/5	50.0%/(1.3, 98.7)/2	33.3%/(0.0, 90.6)/3
Open Ended Lines	Gas	3.6%/(0.4, 12.5)/55	0.0%/(0.0, 11.6)/30	8.0%/(1.0, 26.0)/25
	Light Liquid	5.3%/(3.2, 8.4)/340	2.3%/(0.8, 5.5)/213	10.2%/(5.6, 16.9)/127
Agitator Seals	Gas	0.0%/(0.0, 84.2)/2	0.0%/(0.0, 84.2)/2	-
	Light Liquid	0.0%/(0.0, 97.5)/1	0.0%/(0.0, 97.5)/1	-
Relief Valves	Gas	0.0%/(0.0, 26.5)/12	0.0%/(0.0, 41.0)/7	0.0%/(0.0, 52.2)/5
	Light Liquid	0.0%/(0.0, 33.6)/9	0.0%/(0.0, 41.0)/7	0.0%/(0.0, 84.2)/2
Valves	Gas	7.3%/(5.0, 10.3)/412	2.2%/(0.8, 4.8)/276	17.6%/(11.7, 25.2)/136
	Light Liquid	1.0%/(0.5, 1.7)/1210	0.5%/(0.1, 1.3)/798	1.9%/(0.8, 3.8)/412
Pumps	Light Liquid	10.8%/(4.4, 20.9)/65	9.1%/(2.5, 21.7)/44	14.3%/(3.1, 36.3)/21

Each cell contains: Percent $\geq$ 10,000 ppmv/95% confidence interval/number screened.

SECTION 6

REFERENCES

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4. Sax, N. I. Dangerous Properties of Industrial Materials, 5th Edition. Van Nostrand Reinnold Company, New York, New York, 1979. 1,129 pp.

5. Current Prices of Chemicals and Related Materials. Chemical Marketing Reports, 217 (25): 54-63, 1980.

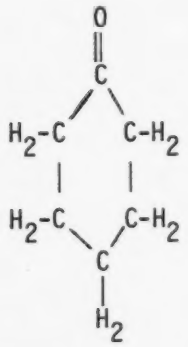
APPENDIX A -- ADIPIC ACID

UNIT 35

SECTION 1

PROCESS DESCRIPTION

Adipic acid is manufactured in this process by the oxidation of a cyclohexanone/cyclohexanol mixture by nitric acid. The chemical reactions are presented below. The cyclohexanone reaction is:

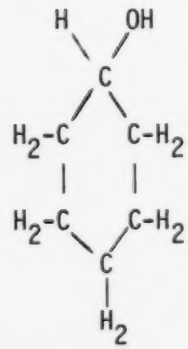


$$+ \quad HNO_3 \longrightarrow \quad H_2\text{-}C\text{-}CH_2\text{-}COOH \quad + \quad NO_x \quad + \quad H_2O \qquad (2\text{-}1)$$

$$H_2\text{-}C\text{-}CH_2\text{-}COOH$$

(Cyclohexanone) (Nitric Adid) (Adipic Acid) (Nitrogen Oxides) (Water)

The cyclohexanol reaction is:



$$+ \quad HNO_3 \longrightarrow \quad H_2\text{-}C\text{-}CH_2\text{-}COOH \quad + \quad NO_x \quad + \quad H_2O \qquad (2\text{-}2)$$

$$H_2\text{-}C\text{-}CH_2\text{-}COOH$$

(Cyclohexanol) (Nitric Acid) (Adipic Acid) (Nitrogen Oxides)(Water)

A simplified process flow plan for the manufacture of adipic acid at this plant is shown in Figure 1. In the oxidation reaction step, a cyclohexanone/cyclohexanol mixture (KA oil) is oxidized to crude adipic acid using nitric acid. The reaction is highly exothermic. Nitrogen oxides off-gas from the reaction is cooled and absorbed in water. The gas that is not absorbed is sent to the boiler for burning under reducing conditions to reduce NO_x emissions. The water stream from the absorber is concentrated and sent to crude adipic acid storage. The waste stream from the concentration step enters a waste tank.

The crude adipic acid stream produced in the reaction is refined in the crystallization and filtration section. The crude adipic acid solution enters a crystallizer where adipic acid is precipitated. The slurry from the crystallizer is filtered. The adipic acid filter cake is dissolved in water and recrystallized. The slurry from the second crystallization is centrifuged.

The wet cake from the centrifuge goes to a dryer where the adipic acid is dried with hot air. The dry adipic acid is cooled and conveyed by a conveyor to a storage hopper. Adipic acid is loaded into hopper cars using a pneumatic conveyer.

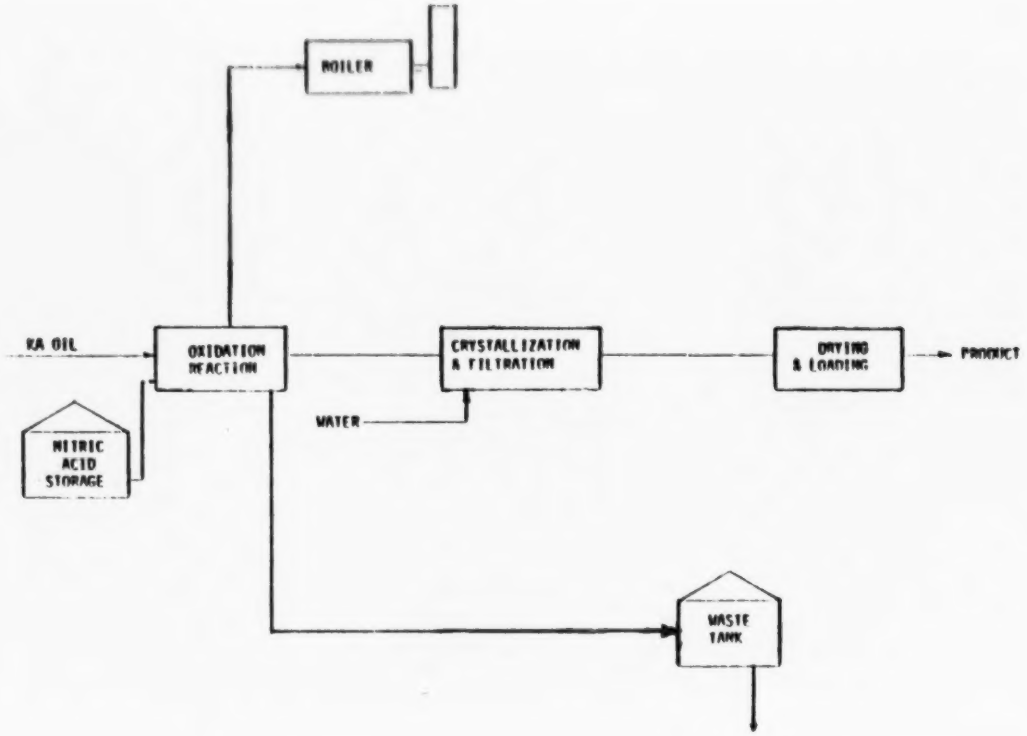


Figure 1. Block Flow Diagram of the Adipic Acid Process Unit Sampled

SECTION 2

SAMPLING PROBLEMS AND OBSERVATIONS

Sampling at the adipic acid unit proceeded in three steps. These steps are:

- Establish unit boundaries
- Screen process sources
- Obtain process information

These steps are discussed below.

2.1 Adipic Acid Unit Boundaries

The adipic acid unit boundaries for the purpose of this study were the battery limits for the unit. These boundaries corresponded to the inlet of KA oil to the oxidation reaction section and end at the exit of "product grade" adipic acid across battery limits as seen on Figure 1.

2.2 Process Sources

Process sources screened at this facility include:

- Valves
- Pump seals
- Process drains
- Relief valves
- Flanges
- Open ended lines

All accessible sources in these groups were screened, except for flanges of which approximately 20 percent were screened. Sources were

considered inaccessible if they could not be reached from a permanent ladder, stairway, or platform.

Initially, the host company had agreed to provide accessibility to all sources. However, since no leaking sources were found in the accessible source population, the host company decided that providing access to the sources normally considered inaccessible would not provide further leak information.

While on-site, the test crew screened an open sample of crude adipic acid (liquid). The sampling instrument registered no response to the compound. Steam was then mixed with the crude adipic acid stream to drive off any volatile organics. The stream was screened again while steam was being introduced. The instrument again recorded no response.

The sampling procedures used throughout this sampling effort correspond to those presented in a detailed sampling plan furnished by the EPA . No problems in either instrumentation or procedure were encountered in this sampling activity.

2.3 Process Information

The process information was assembled by the unit process engineer for each day's screening at the end of that working day. The information was within prescribed ranges when checked against indicators in the process unit.

SECTION 3

SAMPLING TIME

The sampling activity took a total of 35 man-hours to complete. The breakdown of time spent is shown below.

Activity	Time, Man-hours
Instrument calibration and sampling preparation	6
Source sampling	29

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT=35

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	15	0	0	0.00	0	0.00
	LIGHT LIQUID	2	0	0	0.00	0	0.00
	HEAVY LIQUID	218	0	0	0.00	0	0.00
PROCESS DRAINS	HEAVY LIQUID	10	0	0	0.00	0	0.00
OPEN ENDED LINES	GAS	1	3	0	0.00	0	0.00
	LIGHT LIQUID	1	0	0	0.00	0	0.00
	HEAVY LIQUID	102	98	0	0.00	0	0.00
AGITATOR SEALS	HEAVY LIQUID	0	2	0	0.00	0	0.00
RELIEF VALVES	GAS	2	2	0	0.00	0	0.00
	HEAVY LIQUID	0	1	0	0.00	0	0.00
VALVES	GAS	45	3	0	0.00	0	0.00
	LIGHT LIQUID	17	0	0	0.00	0	0.00
	HEAVY LIQUID	1163	69	0	0.00	0	0.00
PUMPS	HEAVY LIQUID	1	59	0	0.00	0	0.00

UNIT 64

PROCESS DESCRIPTION

Process Unit 64 produces adipic acid by hydrogenating phenol to yield a mixture of cyclohexanol and cyclohexanone then oxidizing this mixture. Both reactions use catalysts. The process reactions are:

$$C_6H_5OH + 3H_2 \xrightarrow{Ni} C_6H_{11}OH$$

$$C_6H_5OH + 2H_2 \rightarrow C_6H_{10}O$$

$$\begin{matrix} C_6H_{11}OH \\ C_6H_{10}O \end{matrix} + HNO_3 \xrightarrow[NH_4VO_3]{Cu(NO_3)_2} C_4H_8(COOH)_2 + NO_x + H_2O$$

Four principal reaction steps are involved:

1. Hydrogenation of phenol
2. Oxidation of cyclohexanol/cyclohexanone
3. Stripping of nitrogen oxide
4. Crystallization

The nitrogen oxides generated by reaction step 2 are nitric oxide, nitrogen dioxide, and nitrous oxide. Some organic byproducts are also generated, primarily, glutaric acid, and succinic acid. A flow diagram of the process is shown as Figure 2.

Hydrogenation of Phenol

Cyclohexanol and cyclohexanone are produced by catalytic hydrogenation of phenol. A nickel catalyst is used for cyclohexanol; a palladium catalyst produces primarily cyclohexanone.

Oxidation of Cyclohexanol/Cyclohexanone

Cyclohexanol/cyclohexanone is oxidized in a two-stage process using a 50 percent weight nitric acid solution. The catalysts (cupric nitrate and ammonium metavanadate) are dissolved in the acid.

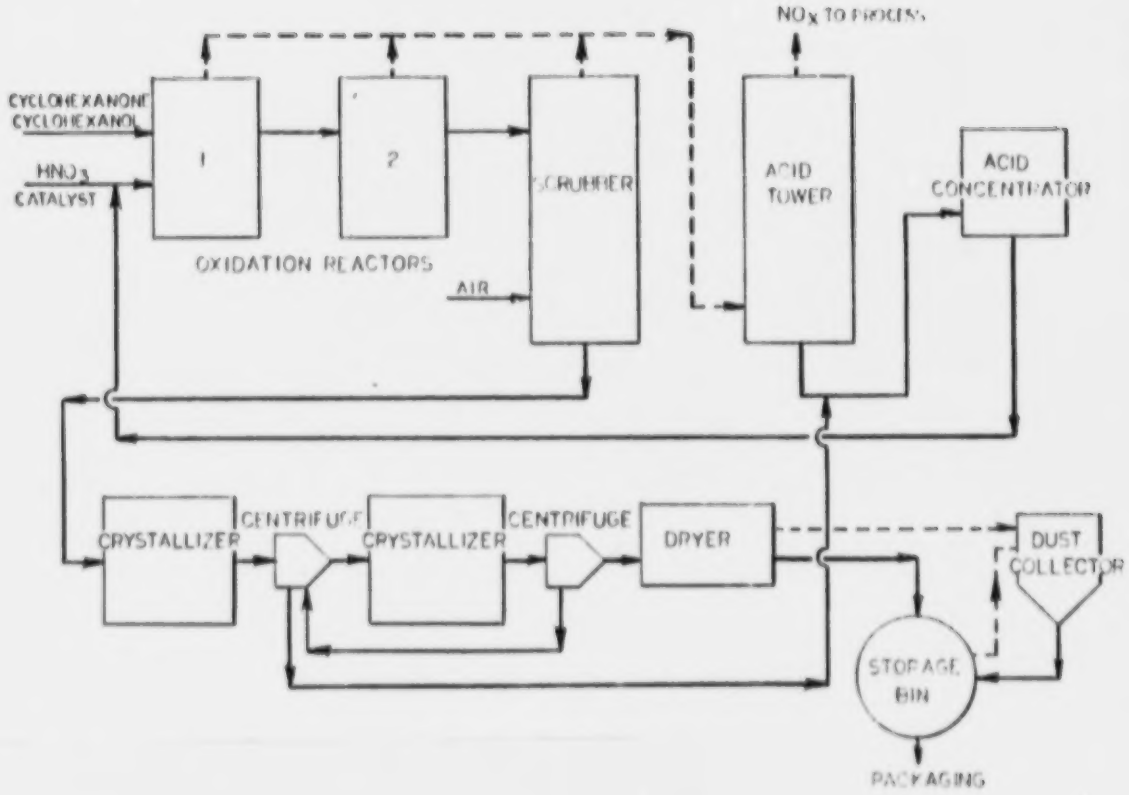


Figure 2. Process flow diagram of adipic acid production.

The nitric acid (containing the catalyst) and cyclohexanol/cyclohexanone streams are fed to the first oxidation reactor in a ratio of about 40:1. The high ratio of nitric acid to cyclohexanol/cyclohexanone is required to complete the oxidation and to provide a sufficient heat sink for the highly exothermic reaction. Reactors for the oxidation are agitators having a pump, heat exchanger, and gas-liquid separator.

The gas-liquid separator removes nitrogen and nitrogen oxides (which are necessary for a smooth, controllable reaction) from the oxidation process. The nitrogen oxides from the separator are oxidized by contact with air and enter an absorber to be recovered as nitric acid. The effluent from the first absorber is further scrubbed in a second absorber. Nitrogen and water vapor, along with some nitric oxide and nitrogen dioxide, are vented from the second absorber.

Stripping of Nitrogen Oxide

The product from the first reactor passes through a preheater and a gas-liquid separator before entering a second oxidation reactor. The reactor effluent then enters a scrubber, in which the dissolved nitrogen oxides are stripped from the adipic acid-nitric acid solution with air. In addition to acting as a stripping agent, the air further oxidizes nitric oxide to nitrogen dioxide ahead of the first absorber.

Crystallization

The adipic acid solution from the scrubber is collected in the adipic acid solution feed tank. Material from the feed tank undergoes vacuum crystallization at 30° to 70°C, followed by centrifugation to remove the mother liquor. Further vacuum recrystallization of adipic acid from water improves the purity of the product. Wet adipic acid from the last crystallization stage is dried, cooled, and transferred to a storage bin. Adipic acid particulates are emitted from the dryer, the cooler, and the product storage bin. The mother liquor from the first crystallization step is recycled to the acid concentrator. The mother liquor stream is continuously purged for removal of other dibasic acids (primarily succinic and glutaric) and for recovery of residual adipic acid, nitric acid, and catalysts.

SAMPLING PROBLEMS AND OBSERVATIONS

Daily calibration of the hydrocarbon detectors was the most important of several quality control procedures. In addition to the morning calibration recommended by EPA, PEDCo conducted a calibration at noon and a calibration drift check every evening. A technique for calibrating the Organic Vapor Analyzer (OVA) Model 128 was adapted for use with the dilution probe. The high standard calibration was adjusted with the pinch clamp regulating the airflow through the diluter. The gas select knob was used to adjust the instrument to the low calibration standard without the use of the dilution probe, with the instrument operating in the 100x mode. For calibrating purposes the low and high calibration standards consisted of methane gas at concentrations of 491 and 8450 ppm.

The repeat screening procedure outlined by EPA was performed on three sources with leaks in different ranges of concentration. The concentration ranges differ from those recommended by EPA, however, because no leaks were found in the high concentration range.

Material coding sheets were completed before entering the plant to facilitate data collection and to minimize the need for communication while collecting data.

The flame-ionization hydrocarbon instrument used in the survey was produced by Century Systems. The OVA-128 with a dilution probe was used. (The OVA-108 had been returned to Century for repairs.) The OVA-128 has a linear readout with a range from 1 to 1000 ppmv. Use of the dilution probe provides capabilities of up to 10,000 ppmv at a 10 to 1 dilution of the sample stream. In this study the OVA-128 was assigned code "2."

Teflon tubing packed with glass wool was used to prevent particulates and liquids from contaminating the OVA probe. Spare tips were carried to replace those that were fouled by particulates. Another problem was unavoidable aspiration of liquid contaminants, which extinguished the hydrogen flame and fouled the probe, we then cleaned and purged the probe and relighted the OVA. When cyclohexanone contaminated the probe, we used an elaborate wash system to purge the OVA.

Operation of the OVA-128 proved to be awkward. Access to the multiplier switch was limited because the cover must be left on to prevent damage to the

instrument. The use of gloves made this problem worse and often led to accidental activation of the wrong switch. Noon and evening calibration checks indicated large drifts, attributable to loosening of the pinch clamp on the dilution probe.

The adipic acid unit had relatively small process lines, making it difficult for the plant escort to distinguish between water lines and product lines. The facility had very few leaks. We tested a number of sources that had solid residue, which indicated possible leaks, but no volatile organic compound (VOC) emissions were measured. A company industrial hygienist took parallel readings with an OVA-128 owned by the company, indicating his interest in the project.

Time was also a factor in increasing the labor-hours required to perform the screening. The PEDCo team had to deliver calibration gas and fuel cylinders by car, lacking time to ship them by freight. Labor-hours were also lost when one member of the screening team needed first aid for a nitric acid burn.

The team tried to screen one of every five flanges. Many sources were not screened, either because they were too high or too difficult to reach safely from a ladder. Other sources could not be sampled because of dripping condensate or nearby process vents.

SAMPLING TIME

Process Unit 64 was a relatively small plant. One team of two persons screened the entire plant in 41 labor hours.

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT #64

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	54	6	0	0.00	0	0.00
	HEAVY LIQUID	102	10	0	0.00	0	0.00
OPEN ENDED LINES	GAS	18	11	0	0.00	0	0.00
	HEAVY LIQUID	112	10	0	0.00	0	0.00
VALVES	GAS	50	11	0	0.00	0	0.00
	HEAVY LIQUID	515	27	0	0.00	0	0.00
PUMPS	HEAVY LIQUID	29	4	1	3.45	0	0.00
COMPRESSORS	GAS	1	0	0	0.00	0	0.00
OTHER[1]	GAS	2	0	0	0.00	0	0.00
	HEAVY LIQUID	1	1	0	0.00	0	0.00

[1]Includes rotary drum seal and tank hatch.

APPENDIX B -- ACETALDEHYDE

UNIT 33

SECTION 1

PROCESS DESCRIPTION

Acetaldehyde is manufactured at this facility (Unit 33) by the direct oxidation of ethylene. The reaction equation is:

$$C_2H_4 + 1/2\ O_2 \xrightarrow[\text{CATALYST}]{} CH_3CHO$$

A simplified flow plan for the process is shown in Figure 1. Ethylene and oxygen are fed to a catalytic reactor. The reaction is exothermic.

The reaction product mixture exits the reactor section and is flashed, separating crude acetaldehyde from catalyst solution and byproducts. Catalyst solution is recycled to the reaction section. The crude acetaldehyde stream is recovered and purified in a series of separation columns.

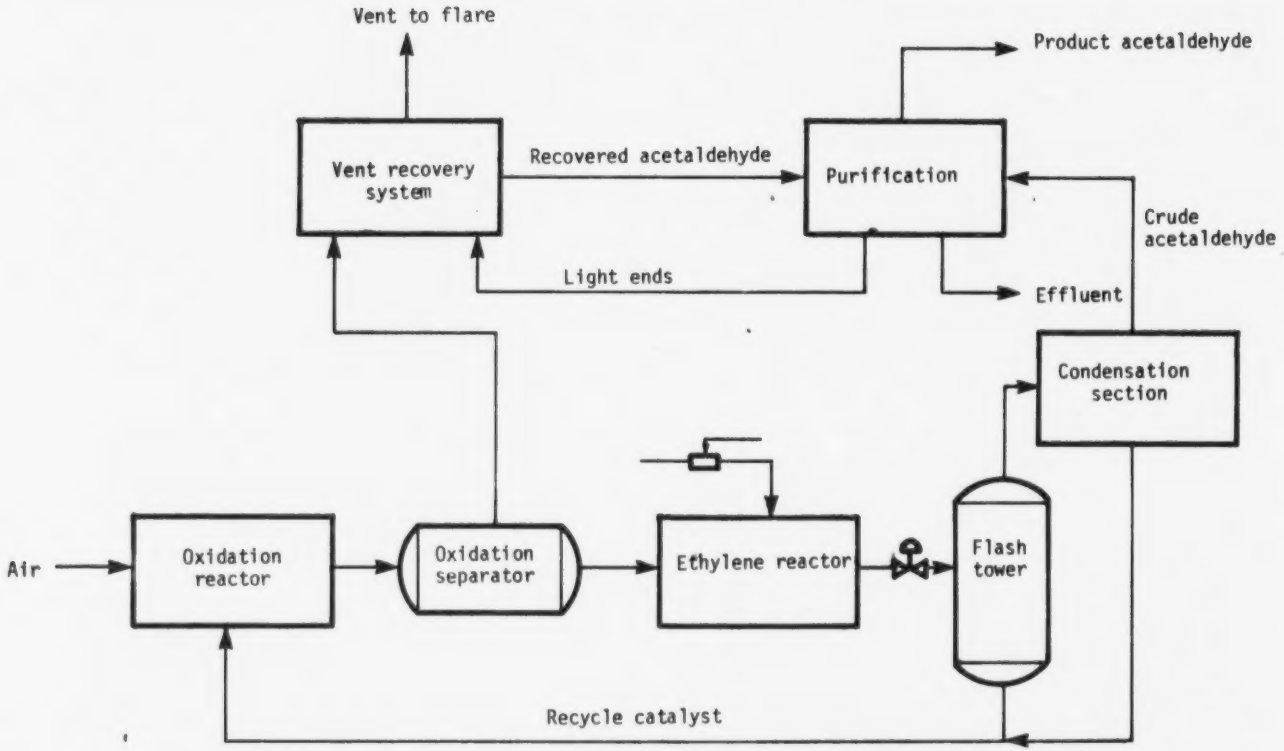


Figure 1. Simplified Process Flow Diagram of Acetaldehyde Process Unit Sampled.

SECTION 2

SAMPLING

Sampling in the acetaldehyde process unit proceeded in three steps. These steps are:

- Establish unit boundaries
- Screen process sources
- Obtain process information

These steps are discussed below.

2.1 UNIT BOUNDARIES

The unit boundaries for the purpose of this study were the battery limits of the process unit. These boundaries correspond to the inlet of ethylene to the reaction and the exit of "product grade" acetaldehyde across battery limits.

2.2 PROCESS SOURCES

Process sources screened at this facility include:

- Valves
- Pump seals
- Process drains
- Relief Valves
- Flanges
- Open ended lines

All accessible sources in these groups were screened, except for flanges, of which approximately 20 percent of the total flange population were screened. Sources were not screened if they could not be reached from a permanent ladder, stairway, platform or a step ladder.

At this process unit, the Acurex screening team was accompanied by a unit operator. The operator numbered each source on piping and instrument diagrams with the corresponding source number on the Acurex data sheet. This information was used to complete the process information sections of the input forms.

Acurex's sampling procedures correspond to those presented in a detailed sampling plan furnished by the EPA. No problems were encountered in the sampling activities.

2.3 PROCESS INFORMATION

The process information was assembled once the screening activity was completed. This information was assembled from mass balances around key process equipment and the numbered sources on plant piping and instrument diagrams. Pressure and temperature data from the mass balance sheets were within the prescribed ranges when compared against indicated readings in the process unit.

SECTION 3

SAMPLING TIME

The sampling activity of this process unit required a total of 45.5 man-hours to complete. The breakdown of the man-hours expended is:

Activity	Time, Man-hours
Instrument calibration and sampling preparation	8.0
Source sampling	37.5

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT=33

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	32	0	0	0.00	0	0.00
	LIGHT LIQUID	144	0	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	12	0	5	41.67	5	41.67
OPEN ENDED LINES	GAS	54	17	4	11.76	3	8.82
	LIGHT LIQUID	158	10	11	6.96	8	5.06
RELIEF VALVES	GAS	4	8	1	25.00	0	0.00
	LIGHT LIQUID	3	0	0	0.00	0	0.00
VALVES	GAS	178	58	10	5.62	8	4.49
	LIGHT LIQUID	551	59	5	0.91	3	0.54
PUMPS	LIGHT LIQUID	32	0	14	43.75	3	9.38

APPENDIX C - Acetone/Phenol
Process Unit No. 12

1. PROCESS DESCRIPTION

This section describes the basic process of process unit #12, acetone/phenol production from cumene. There was considerable discussion in defining the bounds of process unit #12. At the plant site there are three (3) production units that produce acetone and phenol from cumene. These three units operate according to the process technology described as referenced.[1] However, several segments of the three production units operate in common. Therefore, the definition of the process unit required clarification.

Production unit #2 of process unit #12 was chosen for the screening survey for one main reason: Unit #2 is the mid-range in both production capacity and age compared to its sister production units. Most process equipment that was common to all three process units was screened.

The attached figure (1) shows the basic process flow of process unit #12. The purification and cleavage operations consist primarily of three parallel process trains whereas the oxidation and concentration operations are essentially common to the three parallel trains. The oxidation operation consists of two parallel trains, one of which was screened during this survey.

The following list of major process components was screened during the survey:

- cumene unloading
- cumene storage tank
- all oxidizers
- CHP concentration
- decomposer unit
- dehydrator unit
- product cooler unit

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[1] SOURCE ASSESSMENT: Manufacture of Acetone and Phenol from Cumene, EPA-600/2-79-019d. May 1979, Chapter 3.

- acidity removal unit
- crude product storage
- distillation trains (1 of 3)
- batch still
- phenolic water collection/storage
- dephenolizer
- phenate-carbolate springers
- dephenolized H_2O storage tank

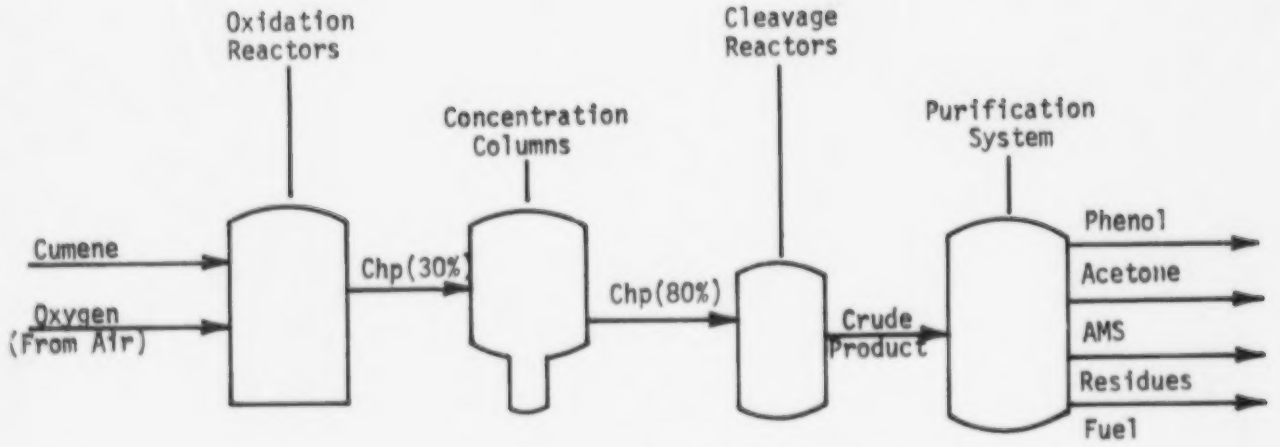


Figure 1. Cumene Phenol Process

2. SAMPLING PROBLEMS AND OBSERVATIONS

The instruments utilized during the screening survey, Century Models OVA-108 and 128, were suspected by plant personnel prior to field testing as possibly being ineffective in measuring several of the major hydrocarbon constituents present on site. Therefore, several checks were performed in the laboratory and in the field. The instrument response for phenol was verified in the laboratory. The instrument probe was placed at the mouth of a bottle containing pure phenol. The temperature of the phenol was approximately 75°F. The OVA-128 showed a low (160 ppm) response to phenol at ambient conditions (20°C and atmospheric pressure). The instrument also took several minutes to rezero after sample introduction. In the field, during a lull in the screening survey due to weather, further response tests were conducted at the request of the host plant. The following table summarizes those testing results for several constituents of importance. All constituents were introduced to the instrument from the gaseous phase above a pure liquid. Phenol and acetophenone required heating to liquify the pure compound before sampling.

OVA RESPONSE TIMES

Constituents	OVA-128 Response	Remarks
Acetone	Normal (2-4 seconds)	20°C gaseous phase above a liquid
AMS	Normal (3-5 seconds)	20°C gaseous phase above a liquid
Cumene	Normal (3-5 seconds)	20°C gaseous phase above a liquid
Phenol	Very Slow (10-30 sec.)[1]	60°C gaseous phase above a liquid
Acetophenone	Slow (8-15 seconds)	60°C gaseous phase above a liquid
Dimethyl-phenol carbinol	No Response	20°C gaseous phase above a liquid

[1] Background zero after sample introduction ~ 2-3 minutes.

Based on this information and realizing the maximum concentrations possible, the OVA-108 and OVA-128 were judged to be adequate screening tools for the purposes of this survey.

The OVA analyzers were operated according to the methods and procedures prescribed by EPA and the manufacturer's instructions. Due to the presence of condensible organics in the vapors leaking from the various components, the OVA analyzer probe was fitted with a glasswool plug to prevent sample line fouling and plugging.

No visible mists or fumes were screened for fear of fouling the detector.

The following unit operations of unit #2 of this facility were not screened for the following reasons:

Units Not Screened	Reason
Oil/Water Separator	Uncovered; no identifiable emission point
Process Residue	Not considered part of process unit
Final Product Storage	Not part of process unit
Loading/Unloading Facility	Not considered part of process unit

Other problems encountered during the screening consisted of:

- instrument operational problems
- weather
- schedule
- unscheduled unit downtime

The instruments, as previously discussed, required extra care in preventing contamination of the detector, due to the nature of several of the compounds screened. The low vapor pressure properties of several compounds resulted in slow and delayed responses. Therefore, extra screening time per component was required. Components with higher than average screening values caused delays, due to slow rezeroing of the instrument.

Bad weather hampered field survey activities on several occasions. The intensity of wind and rain was such that a halt in all screening activity was required for safety reasons on three occasions.

The scheduling of the screening survey around the Easter holidays (due to program constraints) caused a subsequent loss in productivity because of the additional travel time required.

On April 10, 1980, a boiler instrument failure caused a steam generator to shut down. Because the loss of steam supply caused restricted process operations, the TRW crews were withdrawn from the process area until normal operations resumed.

3. SAMPLING TIME

Screening of Process Unit #12 took place during daylight hours between March 27, 1980 and April 11, 1980. Approximately 52 man-days of on-site TRW labor were used by two screening teams consisting of an engineer and technician on each team. Additionally, 15 man-days of host facility labor were expended in providing guidance and assistance to the screening teams. It is estimated that approximately 40 percent of the entire phenol/acetone production unit was covered by the screening survey.

Due to the problems encountered, approximately 9 man-days were lost resulting from either process or instrument unavailability. These estimates exclude time required for travel and preliminary site survey.

Approximately 1 to 1.5 hours per day were spent by each team for the purpose of "set-up". This involved instrument cleaning, calibration and maintenance (if needed) and other preparation. Therefore, approximately 250 man-hours were expended in the process of screening.

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT #12

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	LIGHT LIQUID	92	2	7	6.54	0	0.00
	HEAVY LIQUID	30	0	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	10	0	5	50.00	2	20.00
OPEN ENDED LINES	GAS	2	0	0	0.00	0	0.00
	LIGHT LIQUID	518	64	18	3.47	8	1.54
	HEAVY LIQUID	107	4	0	0.00	0	0.00
RELIEF VALVES	LIGHT LIQUID	19	7	1	5.26	0	0.00
	HEAVY LIQUID	5	0	0	0.00	0	0.00
VALVES	GAS	8	0	0	0.00	0	0.00
	LIGHT LIQUID	1818	257	21	1.16	6	0.33
	HEAVY LIQUID	488	42	1	0.20	0	0.00
PUMPS	LIGHT LIQUID	86	4	6	6.98	2	2.33
	HEAVY LIQUID	36	0	0	0.00	0	0.00

APPENDIX D

ACRYLONOTRILE

PROCESS DESCRIPTION - PLANT A, PROCESS UNIT 65

Plant A uses the Sohio process to produce acrylonitrile by reacting a mixture of propylene, ammonia, and air in the presence of a catalyst. The net process reaction is:

$$C_3H_6 + NH_3 + 3/2\ O_2 \rightarrow CH_2 = CHCN + 3H_2O$$

There is one principal reaction step, the catalytic conversion of propylene to acrylonitrile, followed by purification. Principal byproducts are acetonitrile and hydrogen cyanide. A flow diagram of the process is shown as Figure 1.

Refinery propylene (40 to 90% pure), fertilizer-grade ammonia, and air are fed to a fluidized-bed catalytic reactor. The catalyst is a supported, molybdenum-based catalyst such as 50 to 60 percent bismuth phosphomolybdate on silica. At reaction conditions of 400° to 500°C, 0.5 to 2 atm, and 10 to 20 seconds contact time, the propylene and ammonia are converted to acrylonitrile.

The reactor effluent is fed to an absorption column, where it is scrubbed with water to separate fixed gases and unreacted propylene. The water solution of acrylonitrile from the bottom of the scrubber goes to a separator, where wet acrylonitrile is taken overhead. This product is then dried and purified by distillation. Acetonitrile bottoms from the product separator are purified by conventional distillation methods.

SAMPLING PROBLEMS AND OBSERVATIONS - PLANT A

The most important of several quality control procedures was daily calibration of the hydrocarbon detectors. In addition to the morning calibration

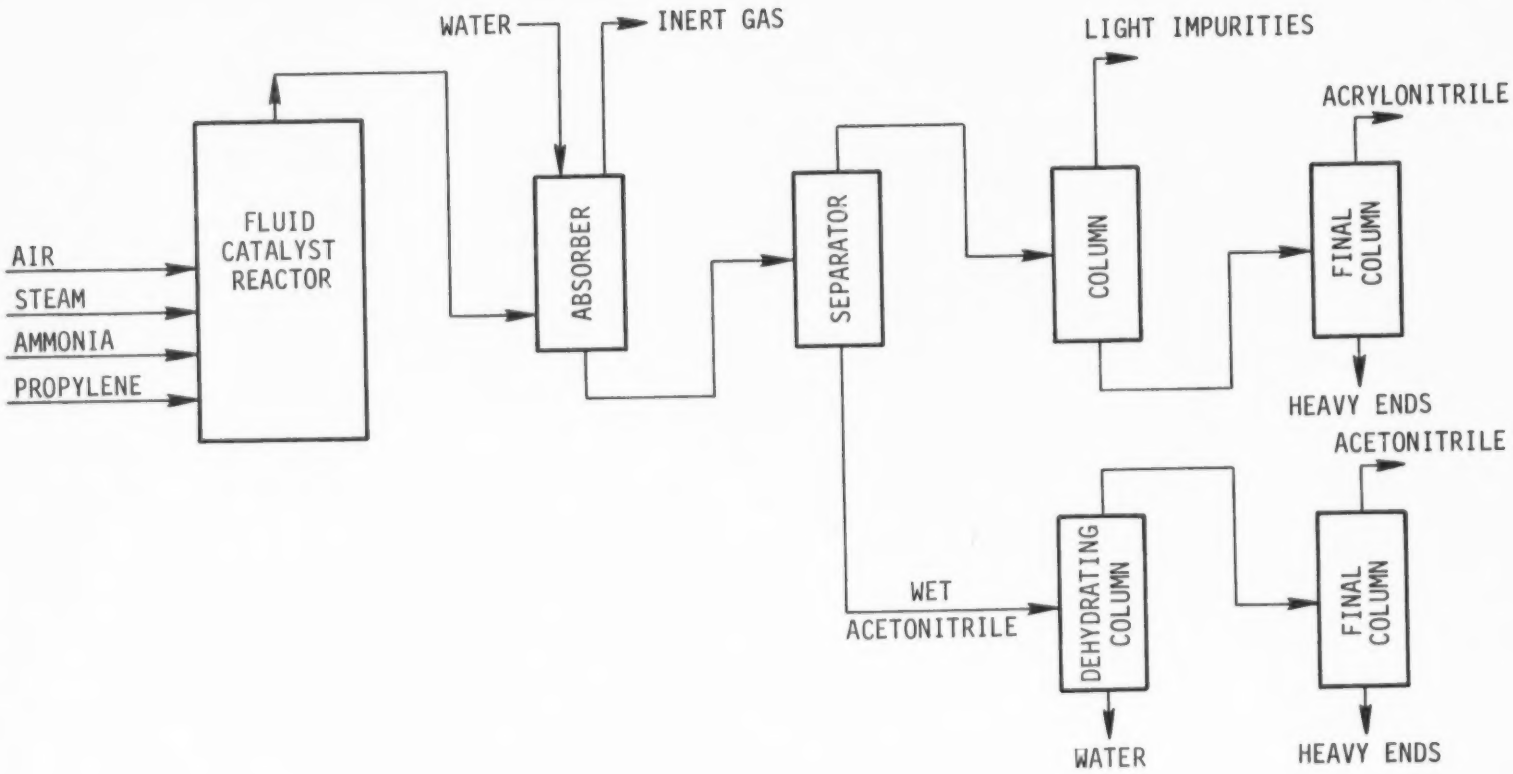


Figure 1. Acrylonitrile process flow diagram - Plants A and B.

recommended by EPA, PEDCo conducted a noon calibration drift check and recalibration and an evening calibration drift check. The procedure for calibrating the Organic Vapor Analyzer (OVA) Model 128 was adapted for use with the dilution probe. The high standard calibration was adjusted with the pinch clamp regulating the airflow through the diluter. The gas select knob was used to adjust the instrument to the low calibration standard without the use of the dilution probe, with the instrument operating in the 100x mode. For calibrating purposes the low and high calibration standards consisted of methane gas at concentrations of 491 ppm and 8450 ppm.

The flame-ionization hydrocarbon instruments used by PEDCo in the survey were produced by Century Systems. The OVA-128 was used with a dilution probe. (The OVA-108 had been returned to Century for repairs.) The OVA-128 has a linear readout with a range from 1 to 1000 ppmv. Use of the dilution probe on the OVA-128 provides capabilities of up to 10,000 ppmv at a 10 to 1 dilution of the sample stream. In this study PEDCo's OVA-128 was coded as "2" and an OVA-128 rented from Digicolor was coded as "4".

The repeat screening procedure outlined by EPA was performed on three sources with leaks in different ranges of concentration. Repeat screenings on the third screening day were performed with only one instrument, because PEDCo's instrument would not leak check nor would the internal electronics adjust to zero. Because of this problem, only one team was able to go into the field the third day; thus several labor-hours were lost. When the instrument was serviced, it was found to have several broken solder connections, a faulty calibrate adjust knob, and a faulty preamplifier.

The PEDCo team designated process unit codes with the help of the host company. Plant A's acrylonitrile unit was designated "65". The material coding sheets were completed before entering the plant to facilitate data collection and to minimize the need for communication while collecting data.

Teflon tubing packed with glass wool was used as a prefilter to prevent particulates and liquids from contaminating the OVA probe. Spare tips were carried to replace those that became fouled by particulates. The dilution probe of the PEDCo OVA-128 (Code 2), however, became clogged with particulates. This problem became apparent when no readout was obtained during screening with the dilution probe in place. The problem was alleviated by cleaning the probe with a cat whisker to eliminate particulates. Another

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT #65

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	75	25	1	1.33	1	1.33
	LIGHT LIQUID	117	13	0	0.00	0	0.00
	HEAVY LIQUID	28	4	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	1	0	1	100.00	1	100.00
	HEAVY LIQUID	1	0	1	100.00	1	100.00
OPEN ENDED LINES	GAS	57	20	1	1.75	0	0.00
	LIGHT LIQUID	160	42	16	10.00	6	3.75
	HEAVY LIQUID	38	10	5	13.16	4	10.53
VALVES	GAS	203	89	11	5.42	9	4.43
	LIGHT LIQUID	591	132	25	4.23	15	2.54
	HEAVY LIQUID	95	19	1	1.05	0	0.00
PUMPS	LIGHT LIQUID	19	5	2	10.53	1	5.26
	HEAVY LIQUID	8	2	3	37.50	2	25.00
COMPRESSORS	GAS	2	0	0	0.00	0	0.00
OTHER[1]	GAS	8	0	1	12.50	1	12.50
	LIGHT LIQUID	2	0	0	0.00	0	0.00
	HEAVY LIQUID	1	0	0	0.00	0	0.00

[1] Includes rupture disk, head gasket, and mixer.

PROCESS DESCRIPTION - PLANT B, PROCESS UNIT 66

Plant B also uses the Sohio acrylonitrile production process. The process catalytically converts propylene to acrylonitrile, which is subsequently purified.

Conversion of propylene is similar to that in Plant A, utilizing the same reaction and equipment. Plant B uses multiple reactors, whereas Plant A had only one. Chemical grade propylene (>90% pure) is used in Plant B instead of refinery grade (40 to 90% pure) used in Plant A.

The purification process used in Plant B is essentially the same as that in Plant A. The reactor effluent is scrubbed with water. This water solution of wet acrylonitrile goes to a separator. The product is then dried and purified by distillation. A flow diagram of the process is shown as Figure 1.

SAMPLING PROBLEMS AND OBSERVATIONS - PLANT B

Quality control procedures were the same as at Plant A, including the morning calibration, noon recalibration, and noon and evening check for calibration drift of the hydrogen detectors. The equipment was operated in the same mode as at Plant A, with the OVA-128 instruments adapted for use with the dilution probe. Again, the low and high calibration standards were methane gas at concentrations of 491 ppm and 8450 ppm.

Repeat screening followed the procedure outlined by EPA. The unit was screened in sections by two teams of two persons. Both teams screened about the same number of emission sources. At company request, all leak points were marked and reported to the unit supervisor.

Coding of the instruments was performed as at Plant A: PEDCo's OVA-128 was coded as "2"; and Digicolor's OVA-128 was coded as "4". Plant B's acrylonitrile unit was coded as "66". The material coding sheet was completed before we entered the plant on the basis of information provided by plant personnel.

Instrument problems were the same as at Plant A. The PEDCo OVA-128 would not leak check on the second day of screening. The needle on the readout meter would not hold steady when the internal electronics were being adjusted to zero. In discussions by telephone, sales representatives of Digicolor in Columbus, Ohio, diagnosed the problem as a broken solder connection. The

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT #66

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	67	9	1	1.49	1	1.49
	LIGHT LIQUID	285	46	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	7	0	1	14.29	0	0.00
OPEN ENDED LINES	GAS	59	9	1	1.69	1	1.69
	LIGHT LIQUID	326	44	9	2.76	6	1.84
VALVES	GAS	193	28	1	0.52	0	0.00
	LIGHT LIQUID	903	190	18	1.99	13	1.44
PUMPS	LIGHT LIQUID	42	0	10	23.81	4	9.52
OTHER[1]	LIGHT LIQUID	2	0	0	0.00	0	0.00

[1]Atmospheric vent.

APPENDIX E

CHLORINATED ETHANES

PROCESS DESCRIPTION, PROCESS UNITS 60, 61, 62

Plant A produces perchloroethylene (Process Unit 60), trichloroethylene (Process Unit 60), 1,1,1-trichloroethane (Process Unit 61), and ethylene dichloride (EDC, Process Unit 62).

Production Of Perchloroethylene and Trichloroethylene

Perchloroethylene and trichloroethylene are produced in a single stage oxychlorination process from ethylene dichloride and chlorine. The net process reaction is:

$$2\ C_2H_4Cl_2 + 1.5\ Cl_2 + 1.75\ O_2 \rightarrow C_2HCl_3 + C_2Cl_4 + 3.5\ H_2O$$

Four principal process steps are involved:

1. Chlorination of ethylene dichloride
2. Phase separation and dehydration
3. Perchloroethylene/trichloroethylene separation
4. Neutralization

Production of perchloroethylene and trichloroethylene takes place in a single process unit. Figure 1 illustrates process flow.

Chlorination of Ethylene Dichloride--

Ethylene dichloride, chlorine, oxygen, and recycled chlorinated organics are fed to a fluid-bed reactor. An inexpensive copper chloride catalyst is used. The multistep reaction that takes place is:

$$2\ C_2H_4Cl_2 + 5\ Cl_2 \rightarrow C_2H_2CL_4 + C_2HCl_5 + 5\ HCl$$

$$C_2H_2Cl_4 + C_2HCl_5 \rightarrow C_2HCl_3 + C_2Cl_4 + 2\ HCl$$

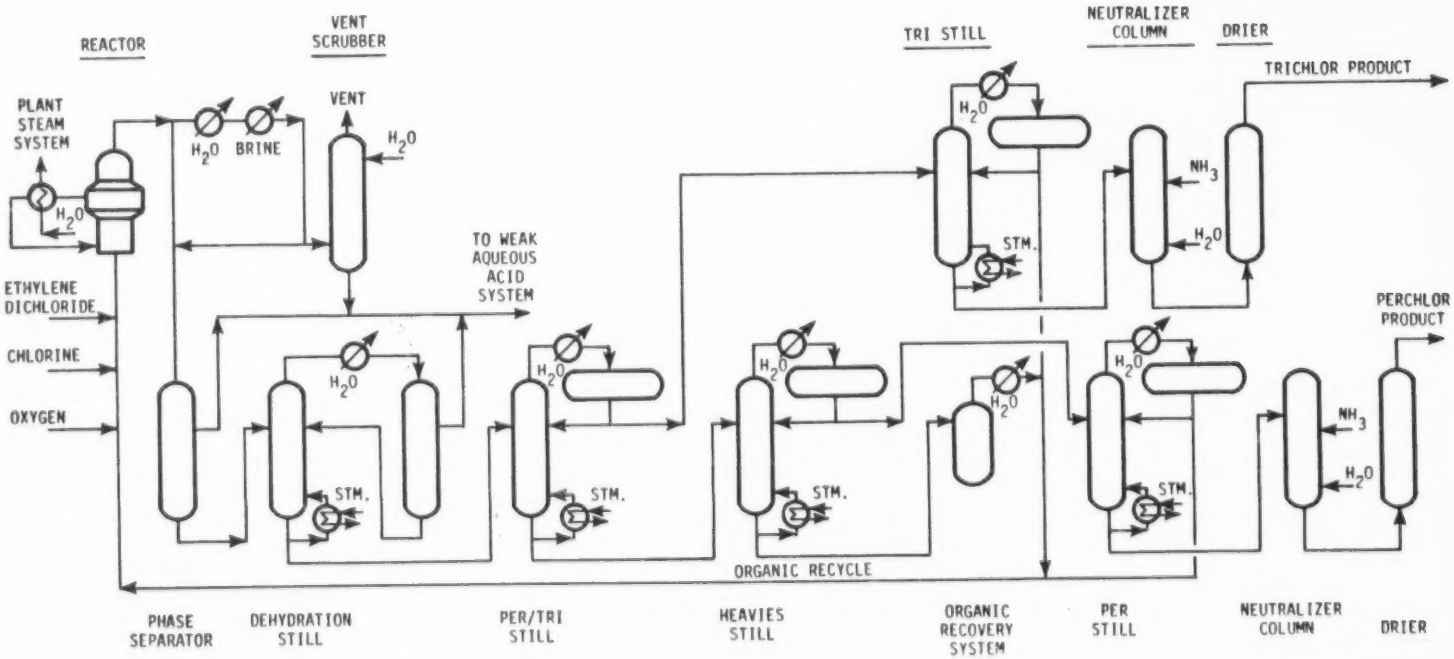


Figure 1. Perchloroethylene/trichloroethylene process flow diagram.

$$7\ HCl + 1.75\ O_2 \rightarrow 3.5\ H_2O + 3.5\ Cl_2$$

The net reaction is highly exothermic. Use of oxygen greatly simplifies product recovery by eliminating noncondensables. The reactor is maintained under pressure and at about 425°C. Product ratios may vary through feed adjustments from all perchloroethylene to nearly all trichloroethylene.

Phase Separation and Dehydration--

After vent scrubbing, the condensed crude and weak acids are phase separated. The crude is dried by azeotropic distillation to produce a noncorrosive still line feed.

Perchloroethylene Trichloroethylene Separation--

The first column (per/tri column or still) splits the crude into two streams, one rich in trichloroethylene and the other in perchloroethylene. The perchloroethylene-rich stream, containing midboilers and heavies, is then fed to the heavies column. High boilers (1,1,2,2- and 1,1,1,2-tetrachloroethane, pentachloroethane, hexachloroethane, dimers, tar, and carbon) are removed as bottoms and flashed to remove tars and carbon. Tars and carbon are incinerated to produce the byproduct muriatic acid. The crude then moves to the perchloroethylene product still, where midboilers are removed overhead. The lights, heavies, and midboilers (consisting of saturated and unsaturated C_2 chlorocarbons) are recycled.

The crude trichloroethylene-rich stream is fed to the trichloroethylene product still, where low boilers, such as dichloroethylenes, are removed overhead and recycled to the reactor. The trichloroethylene product is removed at the bottom.

Neutralization--

The perchloroethylene recovered from the bottom of the perchloroethylene still has a purity >99.9 percent by weight. This product is neutralized with ammonia, washed, and dried. Trichloroethylene from the bottom of the trichloroethylene still also has a purity >99.9 percent by weight, and is also neutralized with ammonia, washed, and dried. Both products are colorless when removed from the still bottom.

Production of 1,1,1-Trichloroethane

An organic compound is chlorinated to produce 1,1,1-trichloroethane (methyl chloroform). The organic compound employed as the raw material for the production of methyl chloroform is confidential information.

Figure 2 illustrates the process employed to produce methyl chloroform. The organic compound and chlorine enter the reactor where hydrogen chloride (HCl), methyl chloroform, and heavies are formed. The HCl is removed from the reactor and purified. The methyl chloroform stream flows to several distillation columns to remove the heavies (byproduct) and to a purification system to remove HCl, water, and other impurities. Appropriate stabilizers are added to the treated methyl chloroform, making the material suitable for commercial use.

Production of Ethylene Dichloride

Ethylene dichloride, used in the production of perchloroethylene and trichloroethylene, is produced in a vapor reaction of ethylene, oxygen, and HCl. The reaction takes place in the presence of a catalyst developed at Plant A. The net reaction is:

$$C_2H_4 + 2\ HCl + \tfrac{1}{2}\ O_2 \rightarrow C_2H_4Cl + H_2O$$

The heat of reaction is recovered as nigh pressure steam and used in other portions of the process. The condensable fraction of the reaction mixture, containing EDC, HCL and water, is removed from the reactor discharge. This fraction is then purified to separate EDC from the remaining materials. The vapor fraction, which includes unreacted ethylene and noncondensable inerts, is recycled to the reactor.

SAMPLING PROBLEMS AND OBSERVATIONS

The most important of several quality control procedures was daily calibration of the hydrocarbon detectors. In addition to the morning calibration recommended by EPA, PEDCo calibrated the Organic Vapor Analyzer (OVA) Model 128 at noon and performed a calibration drift check every evening. The high standard calibration was adjusted with the pinch clamp regulating the airflow through the diluter. The gas select knob was used to

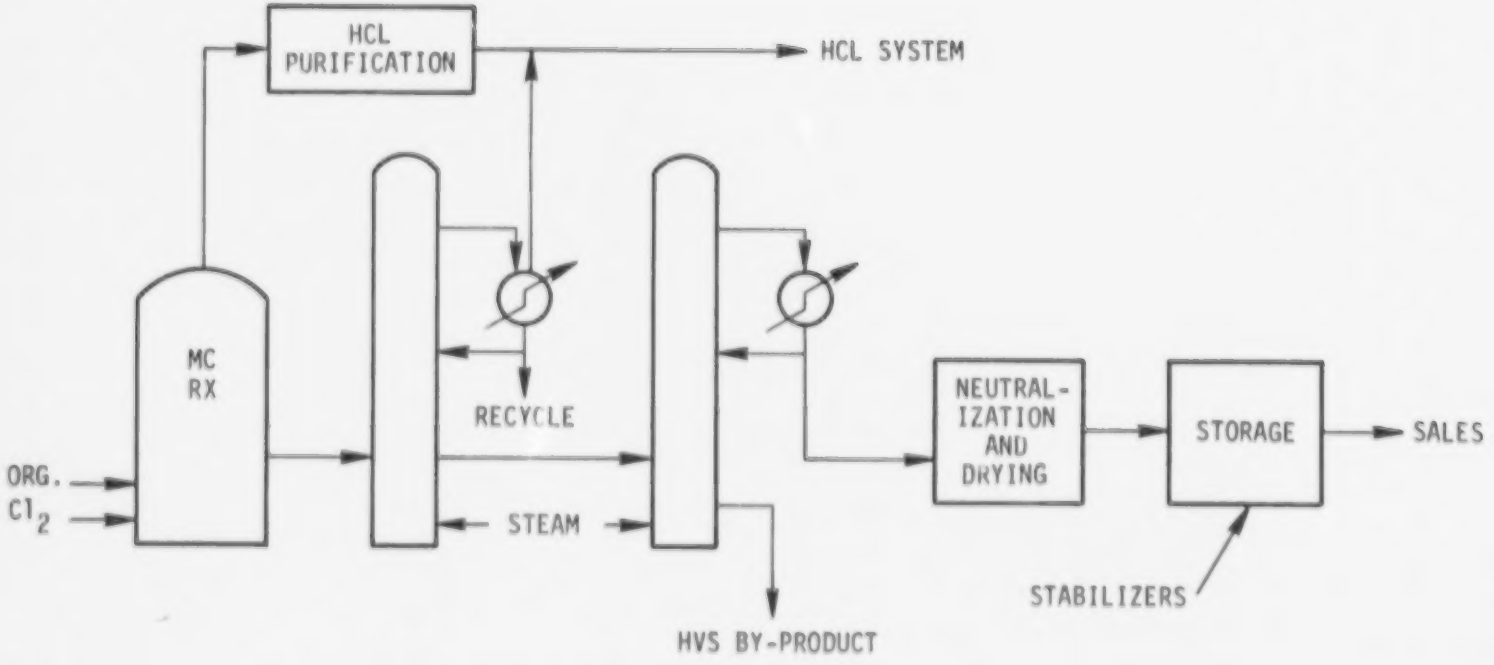


Figure 2. Process flow diagram of 1,1,1-trichloroethane production.

adjust the instrument to the low calibration standard without the use of the dilution probe, with the instrument operating in the 100x mode.

The dilution probe carbon pack was changed every third day to ensure proper operation. The low and high calibration standards consisted of methane gas at concentrations of 491 ppm and 8450 ppm.

The flame-ionization hydrocarbon instruments used by PEDCo in the survey were produced by Century Systems. Two models were used: the OVA-108 and the OVA-128. The only difference between the two models is that the OVA-108 has a log-scale readout with a range from 1 ppmv to 1 percent, whereas the OVA-128 has a linear readout ranging from 1 to 1000 ppmv. Use of the dilution probe on the OVA-108 provides capabilities of up to 100,000 ppmv at a 10 to 1 dilution of the sample stream. The OVA-128 dilution probe dilutes the sample stream to a 10 to 1 dilution, providing the OVA-128 with capabilities of up to 10,000 ppmv.

An instrument defect in the OVA-108 was discovered on the first day of screening. During morning calibration of the instrument, the internal electronic span ranged from 200 ppm to 1000 ppm instead of the expected 10 ppm to 10,000 ppm. The instrument was dismantled, but an attempted repair was unsuccessful. We decided that the instrument needed factory service and could not be used for this screening.

Consequently, the PEDCo team borrowed an OVA-128 from the plant's environmental department. This OVA-128 was coded as "3." PEDCO's OVA-108 was coded as "1." An OVA-128 was rented from Digicolor in Columbus, Ohio, to replace the OVA-108. Digicolor's OVA-128, coded as "4," was brought to the screening site on the third screening day. PEDCo's OVA-128 was coded "2."

Instrument problems were also encountered in the PEDCo OVA-128 (Code 2). The battery pack in the OVA-128 would not hold a charge. On the afternoon of the third screening day, the instrument would not light because the battery pack had gone dead. The dead battery pack was replaced, but the new pack was not charged; thus, the instrument could not be used that afternoon. The OVA-128 rented from Digicolor (Code 4) was taken into the field that afternoon, but the battery pack in that instrument was not fully charged either, and after 1 hour of screening the instrument had a flameout and would not relight. These problems resulted in a loss of labor-hours.

The PEDCo team coded process units with the help of the host company. The plant included three units: the perchloroethylene-trichlorethylene unit was coded as "60," the 1,1,1-trichloroethane unit was coded as "61," and the vinyl (ethylene dichloride) unit was coded as "62." The material coding sheets were completed before entering the plant to facilitate data collection.

Teflon tubing packed with glass wool was used as a prefilter to prevent particulates and liquids from contaminating the OVA probe. Spare tips were carried to replace those that were fouled by particulates. Another problem was the unavoidable aspiration of liquid contaminants, especially around pump and compressor seals. These contaminants usually extinguished the hydrogen flame, necessitating additional time to purge and relight the OVA.

Repeat screenings were performed as outlined by EPA when practical. Only one repeat screening source was found the first day. Sources found the second day were screened in addition to the one from the previous day. One repeat screening source found the second day was in a construction area the third day, and therefore could not be screened. A repeat screening of one source could be not be performed on the fourth day because HCl was leaking nearby. This source was replaced with another source in the same range.

Operation of the OVA-128 proved to be awkward. Access to the multiplier switch was limited because the cover must be left on to prevent damage to the instrument. The use of gloves while climbing made this problem worse, and often led to accidental activation of the wrong switch.

Screening of the perchloroethylene-trichlorethylene, 1,1,1-trichloroethane, and ethylene dichloride units was arranged to facilitate data collection and to prevent duplication of screening efforts. First, both screening teams covered the perchloroethylene-trichloroethylene process unit in its entirety. One team screened the chlorinator in the 1,1,1-trichloroethane unit while the other team began screening the ethylene dichloride.

Overall, the screening program went relatively smoothly in spite of several problems that slowed progress. Time was an important factor. The PEDCo team had to deliver calibration gas and fuel cylinders by car, lacking time to ship them by freight. Additional time was needed to complete the screening of all three process units. Instrument problems caused a loss of labor-hours. High temperatures combined with high humidity made it inadvisable to do any strenuous climbing in the afternoons.

The teams tried to screen one of every five flanges. Many sources were not screened either because they were too high or too difficult to reach safely. Occasionally, other sources could not be sampled because of dripping liquid, which prevented use of the instrument.

Plant supervisors checked our data sheets and discovered some discrepancies in the primary material identifications. A PEDCo team met with representatives of the plant to discuss the coding and to clarify all discrepancies.

SAMPLING TIME

Two teams of two persons each screened the plant in 180 labor-hours.

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT A60

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	11	11	0	0.00	0	0.00
	LIGHT LIQUID	525	3	0	0.00	0	0.00
	HEAVY LIQUID	2	0	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	9	0	2	22.22	0	0.00
OPEN ENDED LINES	GAS	10	11	0	0.00	0	0.00
	LIGHT LIQUID	412	71	15	3.64	6	1.46
	HEAVY LIQUID	4	0	0	0.00	0	0.00
RELIEF VALVES	LIGHT LIQUID	5	2	0	0.00	0	0.00
VALVES	GAS	28	24	0	0.00	0	0.00
	LIGHT LIQUID	1620	180	14	0.86	1	0.06
	HEAVY LIQUID	12	0	0	0.00	0	0.00
PUMPS	LIGHT LIQUID	60	0	16	26.67	5	8.33
OTHER[1]	GAS	3	2	1	33.33	1	33.33
	LIGHT LIQUID	21	6	3	14.29	2	9.52

[1]Includes expansion joint, rupture disk, and sight glass seal.

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT #61

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	0	3	0	0.00	0	0.00
	LIGHT LIQUID	73	6	1	1.37	0	0.00
OPEN ENDED LINES	LIGHT LIQUID	111	19	5	4.50	2	1.80
AGITATOR SEALS	LIGHT LIQUID	1	0	0	0.00	0	0.00
VALVES	LIGHT LIQUID	373	57	10	2.68	4	1.07
PUMPS	LIGHT LIQUID	10	0	4	40.00	1	10.00
OTHER[1]	LIGHT LIQUID	2	0	0	0.00	0	0.00

[1]Rupture disk.

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT #62

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	5	4	0	0.00	0	0.00
OPEN ENDED LINES	GAS	17	5	0	0.00	0	0.00
VALVES	GAS	20	9	0	0.00	0	0.00

APPENDIX F - Cumene

Unit 5

1. PROCESS DESCRIPTION

Process unit number five is a cumene manufacturing facility. The manufacturing process is shown in Figure 1. Propylene (in a propylene/propane mixture) and benzene are reacted at elevated temperatures and pressures in the presence of a catalyst to form an unrefined cumene product. Product purification occurs in a three-tower fractionating system. Propane is taken as an overhead vapor and returned to the propylene/propane supplier. Excess benzene is recycled back to the reactor, and cumene is produced as an overhead stream. The residual bottoms are sent to a slop oil recovery system.

The entire process unit was screened, from feed of benzene and propylene/propane to the unit through delivery of the cumene product, recovered propane, and residual bottoms from the unit.

2. SAMPLING PROBLEMS AND OBSERVATIONS

Less than 10 percent of the 550 process valves identified were not screened. Most of these unscreened valves could have been accessed by using a ladder. The remaining nine unscreened valves were located in upper tiers of pipe racks or in difficult-to-access places high on fractionating columns. Screening of these nine valves would have required a major effort (such as the use of extensive scaffolding, a crane, or a "cherry picker").

Nine of the 10 relief valves in this unit were connected directly to a flare header. Gaining access to these relief valves' vents for screening was therefore not possible. The relief valve servicing the depropanizer column overhead (mostly propane and propylene) was vented directly to the atmosphere, and it was screened.

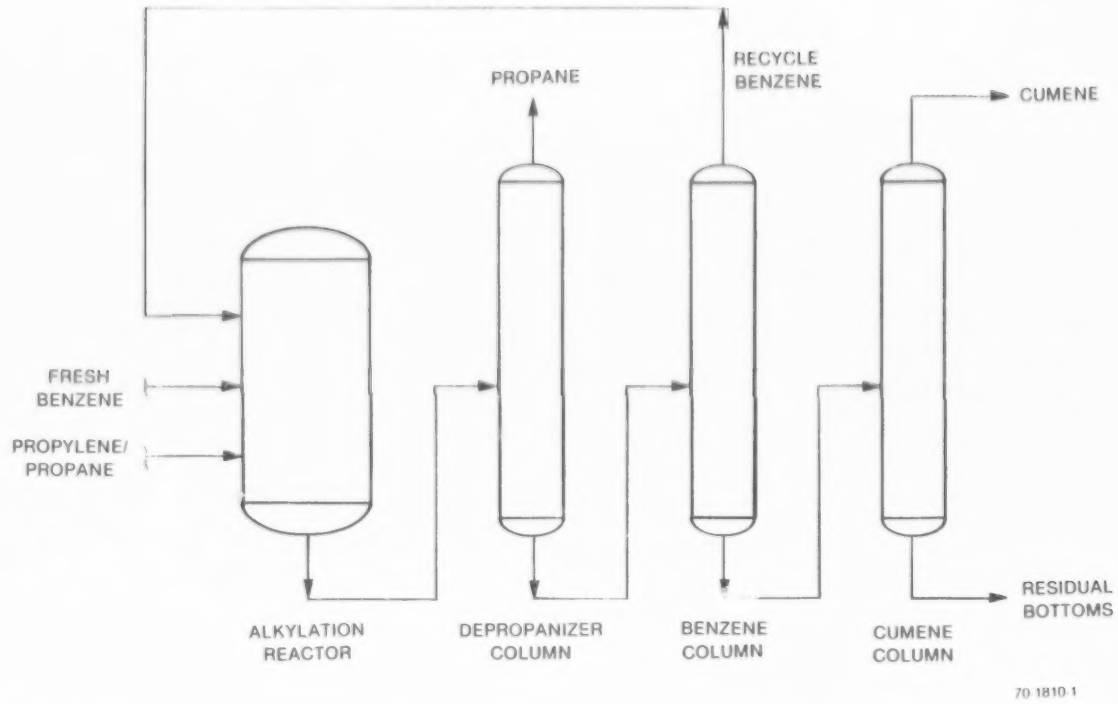


FIGURE 1 PROCESS FLOW DIAGRAM - UNIT 5 (CUMENE)

All 11 pumps in the cumene process unit had single mechanical seals. These pumps were screened directly at the seal area.

3. SAMPLING TIME

The screening program at process unit number five was conducted from June 2 to June 5, 1980. The actual screening effort, including calibration, required 32 person-hours by a two-person team. An additional 24 person-hours were expended for plant and safety orientation, set-up and removal of screening equipment, and acquisition of process data.

Approximately 1,080 potential fugitive emission sources were identified. However, only about 1,020 of these sources were actually screened. Screening of these sources required 16 hours by the two-person team (32 person-hours), an average of about 0.9 minute per source (or about 1.9 person-minutes per source).

4. DATA SUMMARY

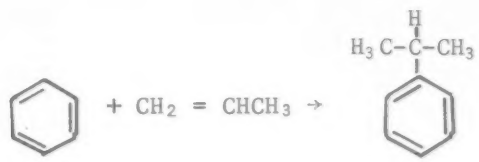
SUMMARY STATISTICS
UNIT=5

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	100	0	2	2.00	1	1.00
	LIGHT LIQUID	317	0	8	2.52	1	0.32
	HEAVY LIQUID	58	0	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	31	0	3	9.68	1	3.23
OPEN ENDED LINES	GAS	2	0	0	0.00	0	0.00
	LIGHT LIQUID	6	0	0	0.00	0	0.00
RELIEF VALVES	GAS	1	5	1	100.00	1	100.00
	HEAVY LIQUID	0	4	0	0.00	0	0.00
VALVES	GAS	108	15	15	13.89	5	4.63
	LIGHT LIQUID	328	26	66	20.12	36	10.98
	HEAVY LIQUID	63	8	1	1.59	0	0.00
PUMPS	LIGHT LIQUID	10	0	8	80.00	3	30.00
	HEAVY LIQUID	1	0	0	0.00	0	0.00

Unit 6

1. PROCESS DESCRIPTION

Process unit number six is a cumene manufacturing facility. The manufacturing process is shown in Figure 2. Propylene (in a propylene/propane mixture) and benzene are catalytically reacted at elevated temperatures and pressures to form a crude cumene product, according to the following simplified reaction:



(Benzene) (Propylene) (Cumene)

The reactor effluent is depropanized in the rectifier tower. Excess benzene is then removed as overhead from the benzene tower and recycled back to the reactor. The refined cumene product is distilled from the residual bottoms in the cumene tower.

The entire process unit was screened, from feed of benzene and propylene to the unit through delivery of the cumene product, recovered propane, and residual bottoms from the unit. In addition, process information was gathered for two propylene/propane compressors upstream of the cumene manufacturing process.

2. SAMPLING PROBLEMS AND OBSERVATIONS

Approximately 20 percent of the 1,170 process valves identified in process unit number six were not screened. Most of these unscreened valves could have been accessed by using a ladder. The remaining 60 unscreened valves were located in upper tiers of pipe racks or in difficult-

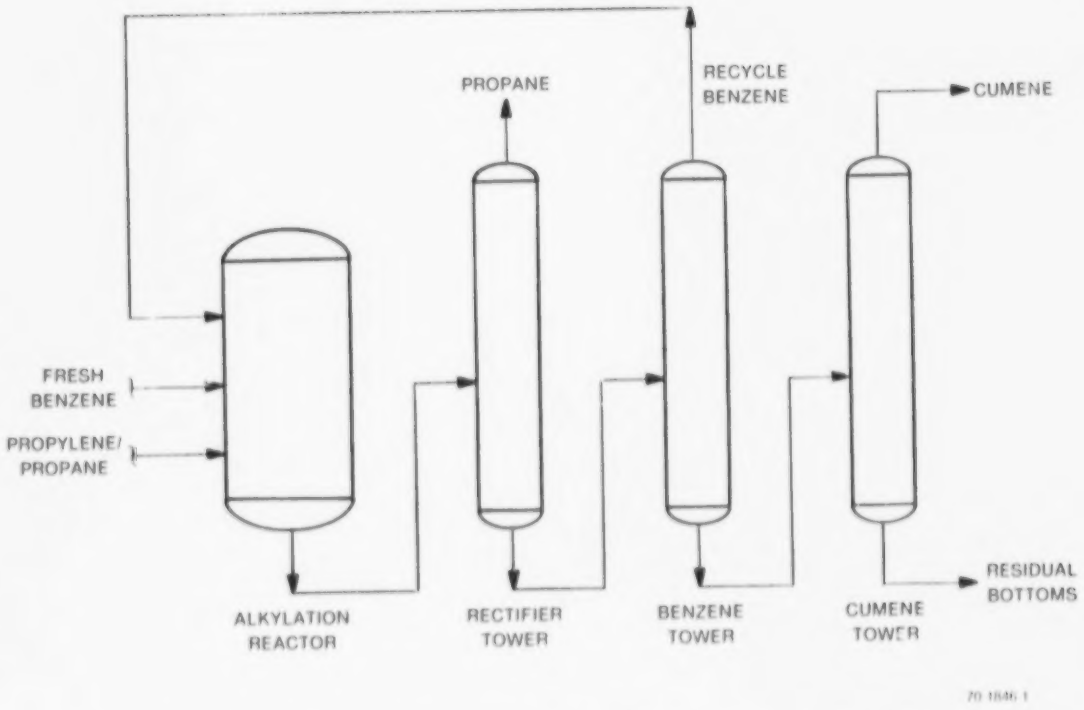


FIGURE 2 PROCESS FLOW DIAGRAM — UNIT 6 (CUMENE)

to-access places high on fractionating columns. Accessing these valves for screening would have required a major effort, such as using extensive scaffolding, a crane, or a "cherry picker".

Seventeen of the 18 pressure relief valves in the unit vented directly to the flare header. One relief valve vented back to the process. Therefore, the vents for these relief valves were not accessible for screening.

The seal areas of the two propylene/propane compressors were enclosed and vented to the flare. The single mechanical seals were therefore not accessible for screening.

All 15 pumps in process unit number six were equipped with tandem dual mechanical seals. Two pumps had inboard and outboard seals, so 17 seals were screened. All pumps were screened directly at the outer seal of the dual mechanical seals. Detailed information about the pump seals is presented in Table 1.

The host company had conducted a fugitive source maintenance program during the two weeks prior to our screening. This was said to be part of a regular maintenance program conducted on approximately a quarterly basis. A J. W. Bacharach explosimeter, with a readout in percentage of the lower explosive limit, was used for leak detection and directed maintenance. No details on the effectiveness of the maintenance were obtained since sources were maintained as soon as they were detected, with little or no record keeping involved.

The Century Systems OVA-108 used in the screening described in this report displayed sluggish response to fugitive emissions of aromatics, particularly cumene. When the instrument probe was introduced to an aromatic concentration at a leaking source, a fairly long period of time elapsed before the instrument readout stabilized at a maximum concentration. In addition, the instrument's recovery from the maximum concentration to the ambient, or background, concentration was very slow.

TABLE 1. PUMP SEAL DATA - UNIT 6

Source ID Number	Pump Status During Screening	Screening Value, ppmv	Seal Type	Seal Manufacturer	Seal Screening Accessibility	Barrier Fluid Type	Barrier Fluid Pressure	Seal Use History
91	On-line	20	Dual Mechanical (tandem)	John Crane	Screened directly at outer seal	Heavy lubricating oil (static)	9 psig[1]	New[2]
92	Off-line	0	"	"	"	"	"	"
109	Off-line	0	"	"	"	"	"	"
110	On-line	0	"	"	"	"	"	"
1017	On-line	10	"	"	"	"	"	"
1026	Off-line	10	"	"	"	"	"	"
1078	On-line	20	"	"	"	"	"	"
1083	Off-line	0	"	"	"	"	"	"
1232	Off-line	0	"	"	"	"	"	"
1257	On-line	20	"	"	"	"	"	"
1282	On-line	130	"	"	"	"	"	"
1407	Off-line	0	"	"	"	"	"	"
1415	On-line	0	"	"	"	"	"	"
1451	Off-line	0	"	"	"	"	"	"
1456	Off-line	0	"	"	"	"	"	"
1472	Off-line	0	"	"	"	"	"	"
1477	On-line	20,000	"	"	"	"	"	"

[1]A pressure sensor activates an alarm if the barrier fluid pressure exceeds the set pressure of 9 psig. The pump is shut down for repair and a standby pump is brought on-line.

[2]All pumps were fitted with tandem dual mechanical seals at unit start-up in September 1978. Approximately 10 seals were replaced during start-up and during the first year of unit operation. Only two seals had been replaced in the seven months prior to screening.

The instrument used for screening had to be calibrated at frequent intervals because of a varying hydrogen flow rate to the combustion chamber of the instrument's flame ionization detector. Hydrogen flow rate, as indicated by the regulator discharge pressure, appeared to decrease with hydrogen cylinder pressure or time of instrument operation.

3. SAMPLING TIME

Screening of process unit number six was conducted from July 29 to August 1, 1980. The screening effort, including calibration and instrument repair, required 27 hours by a two-person team, or 54 person-hours.

Approximately 1,830 sources were identified, although only 1,570 of these were actually screened. Sources were screened at an average rate of about 1.0 minute per source (or about 2.1 person-minutes per source).

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT #6

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	267	5	27	10.11	18	6.74
	LIGHT LIQUID	251	3	16	6.37	8	3.19
	HEAVY LIQUID	72	0	0	0.00	0	0.00
PROCESS DRAINS	GAS	6	0	1	16.67	0	0.00
OPEN ENDED LINES	GAS	4	0	0	0.00	0	0.00
	LIGHT LIQUID	9	1	3	33.33	2	22.22
	HEAVY LIQUID	1	0	0	0.00	0	0.00
RELIEF VALVES	GAS	0	15	0	0.00	0	0.00
	LIGHT LIQUID	0	3	0	0.00	0	0.00
VALVES	GAS	340	82	102	30.00	58	17.06
	LIGHT LIQUID	471	102	98	20.81	48	10.19
	HEAVY LIQUID	135	42	0	0.00	0	0.00
PUMPS	LIGHT LIQUID	15	0	1	6.67	1	6.67
	HEAVY LIQUID	2	0	0	0.00	0	0.00
COMPRESSORS	GAS	0	2	0	0.00	0	0.00

APPENDIX G - Ethylene
Process Unit No. 11

1. PROCESS DESCRIPTION

This ethylene unit was designed to produce 500 MM #/yr of 99.9% purity ethylene from a feedstock of approximately 65% propane, 30% ethane, 5% butane and methane. In addition to ethylene, the products from the unit include hydrogen, propylene, a mixed C_4 stream, and light and heavy aromatic distillates. Two tail gas streams containing most of the methane recovered in the unit are combined and used as fuel. A light oil stream, a quench residue and an acid gas stream are disposed of offsite.

A generalized flow diagram, Figure 1, is attached as reference. The screening team defined the ethylene facility as all process equipment operating at the time of the survey within the boundaries of the access roads on the plant supplied plot plan. This facility was undergoing a major expansion project of the ethylene facility at the time of the survey. With the exception of one furnace at the northwest corner of the unit boundary, the new process equipment from this expansion was out of service. The tank farm located south of the ethylene unit containing raw materials and final product was not included within the process unit as it was defined and screened.

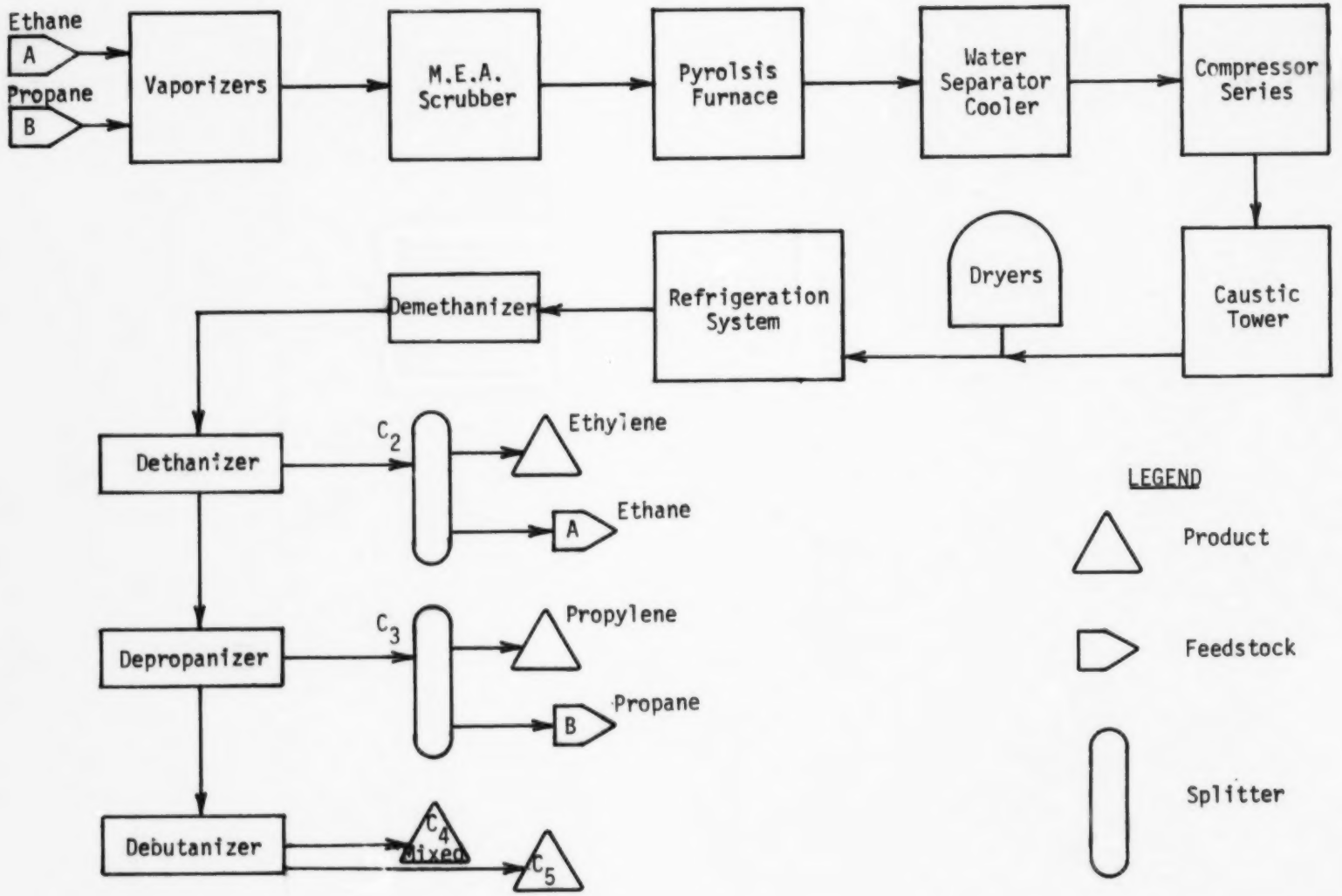


Figure 1. Generalized Process Flow Diagram

2. SAMPLING OBSERVATION AND PROBLEMS

Process Unit #11 was screened by one team consisting of an engineer and a technican. Due to preparations for a forthcoming major process turnaround, there were a number of sources not screened. A large number of these components were gate valves located in overhead pipe racks which were obstructed by construction materials.

The OVA analyzer developed several operational problems during the screening survey, which required trouble shooting and service while at the plant. All problems were resolved by screening team members with the assistance of host plant personnel. On two occasions, components on lines which were being air purged were screened resulting in instrument failure. Viscous hydrocarbon liquid had been forced out through valve stems and entered the probe of the OVA analyzer. The analyzer then required partial dismantling and cleaning.

The screening was conducted during periods when the weather was bitterly cold with substantial wind chill conditions. These harsh weather conditions necessitated frequent breaks during the daily screening survey. There was, therefore, a subsequent loss of productivity.

3. SAMPLING TIME

Screening of process unit #11 took place during daylight hours between February 21, 1980 and March 14, 1980. Approximately 1.5 hours per day were required for preparation (calibration, maintenance, etc.) and clean-up of the OVA analyzer.

Excluding travel time, approximately 34 man-days (17 team days) were spent on site by the TRW team. At least 17 man-days time were spent by the host facility employees in assisting the screening effort.

Approximately 5 man-days of screening time were lost due to weather (snow) and instrument (fouling) problems. Therefore, the screening time was effectively 188 man-hours (excluding host facility time).

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT #11

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	37	2	4	10.81	4	10.81
	LIGHT LIQUID	21	0	1	4.76	1	4.76
	HEAVY LIQUID	1	0	0	0.00	0	0.00
PROCESS DRAINS	GAS	6	1	0	0.00	0	0.00
	LIGHT LIQUID	13	0	5	38.46	4	30.77
OPEN ENDED LINES	GAS	184	24	18	9.78	13	7.07
	LIGHT LIQUID	122	22	38	31.15	20	16.39
RELIEF VALVES	GAS	51	2	7	13.73	2	3.92
	LIGHT LIQUID	11	1	1	9.09	1	9.09
VALVES	GAS	2171	225	404	18.61	221	10.18
	LIGHT LIQUID	995	123	365	36.68	178	17.89
	HEAVY LIQUID	39	20	0	0.00	0	0.00
PUMPS	LIGHT LIQUID	27	0	14	51.85	4	14.81
COMPRESSORS	GAS	7	0	0	0.00	0	0.00

Unit 4

1. PROCESS DESCRIPTION

Process unit number four is an olefins manufacturing unit. Feedstocks, consisting of ethane, naphtha raffinate, and desulfurized gas oil, are steam cracked into a wide boiling range mixture of olefins, diolefins, aromatics, and other hydrocarbons. Separation facilities recover ethylene and propylene along with crude streams of butylene/butadiene, isoprene, gasoline fractions, and by-product gas and liquid fuels, which are recovered in adjacent units. A process flow diagram is shown in Figure 2.

Screening was concentrated on the compression, acid gas removal, refrigeration, and separation systems downstream of the cracking furnaces and quench section (The cracking furnaces and quench section were not considered part of the ethylene process by the plant owners.). Ethylene and propylene product streams were screened through pipeline delivery from the unit. The heavier product streams were screened through the debutanizer column.

The physical size of the facility necessitated limiting the scope of the unit definition to minimize the screening effort. For example, the cracking furnaces were not included within the definition of the light olefins unit. Also, only one of the two parallel streams in dual-train sections within the unit was screened. Hydrogen and fuel gas (methane) systems were not screened.

2. SAMPLING PROBLEMS AND OBSERVATIONS

Approximately 760 of the 4,800 process valves identified were not screened. About 70 percent of these unscreened valves could have been accessed by using a ladder or scaffolding. Screening of the remaining 30 percent would have required a major effort, because these valves were located in upper tiers of pipe racks, in difficult-to-access places high on fractionating columns, in the path of escaping process steam, etc.

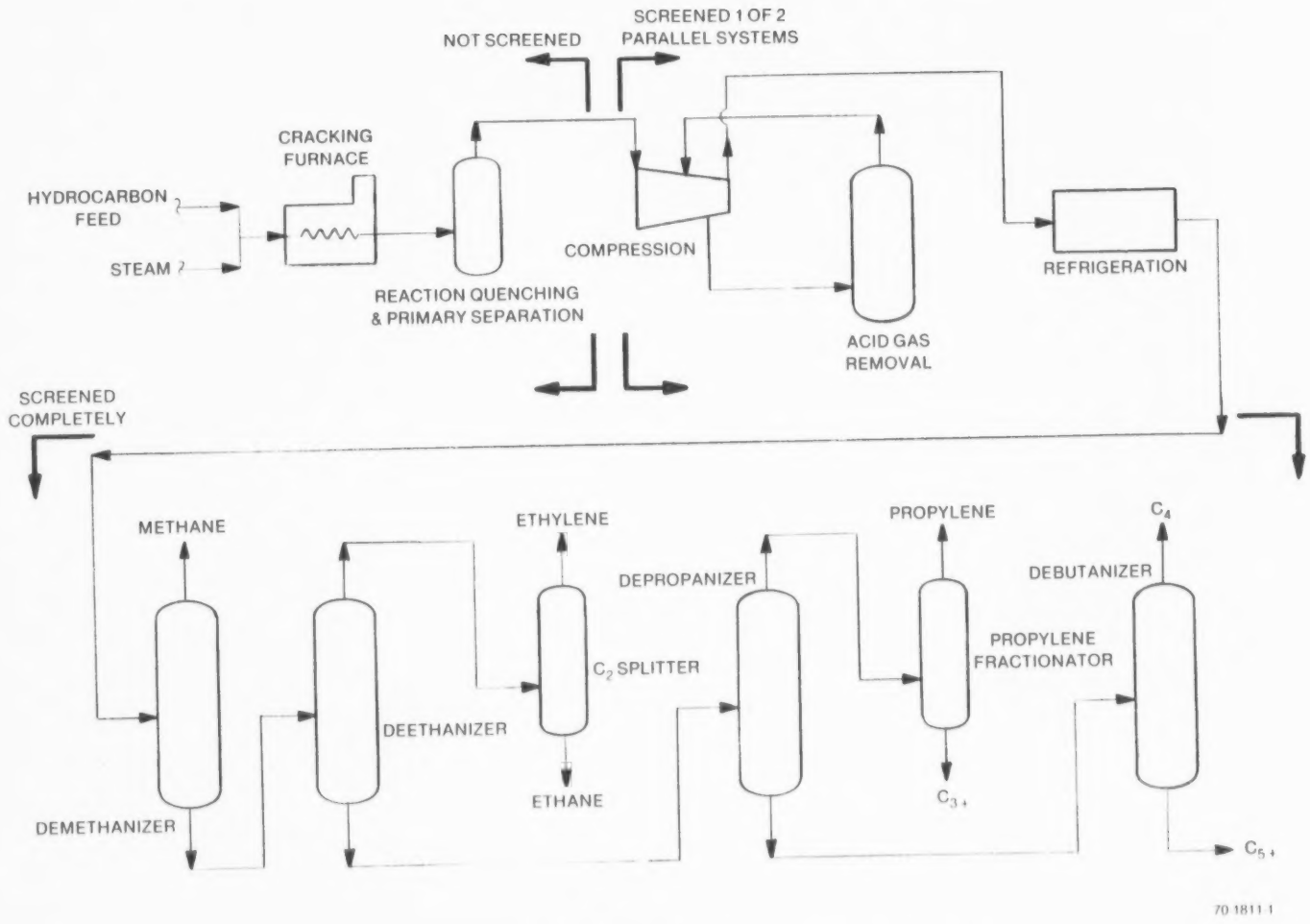


FIGURE 2 PROCESS FLOW DIAGRAM - UNIT 4 (ETHYLENE)

Many of the process valves in cold service were partially covered with ice. Fugitive emissions from these valves were often difficult to detect, as the leak often surfaced at random locations around the ice cap. Also, accurate screening values for these ice-covered valves were difficult to obtain; care had to be taken to prevent moisture from being drawn from wet surfaces near leak areas into the instrument probe.

The 101 relief valves were vented directly to a flare header, rather than to the atmosphere, so hydrocarbon leaks through the relief valve seats would have been burned in the flare. Therefore, the relief valves, although physically accessible, were not screened.

About 80 percent of the 35 pumps screened in process unit number four were screened at the seals, all of which were mechanical. Safety shrouds around the pump shafts or process steam jetting over the seal areas prevented screening of six pump seals.

Fugitive emissions from compressor seals were vented to the flare header or routed back to the suction side of the compressors. Since the seal areas were enclosed, the three compressors were screened at the flange separating the compressor housing from the shroud enclosing the drive shaft.

Approximately 25 man-hours were lost from the screening effort because of instrument malfunctions. Problems included calibration drift and erratic ignition of the hydrogen/air mixture in the flame ionization detector.

3. SAMPLING TIME

Screening of process unit number four required 16 working days from April 18, 1980 until May 12, 1980. A two-person screening team was in operation for the entire time, and they were joined by a second two-person team for three working days.

Almost 6,200 potential fugitive emission sources were identified in those portions of the process unit discussed in Section 1. However, only 5,300 of these sources were actually screened. The total time spent for screening of these sources, calibrating the instruments, and repairing malfunctioning instruments was 128 hours (96.5 hours, 19 hours, and 12.5 hours, respectively), or 256 person-hours. Including time spent for instrument calibration and repair, source screening required an average of about 1.4 minutes per source for a two-person team (or about 2.9 person-minutes per source).

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT 64

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	448	1	48	10.74	30	6.70
	LIGHT LIQUID	335	0	43	12.84	23	6.87
	HEAVY LIQUID	15	0	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	300	0	0	0.00	0	0.00
OPEN ENDED LINES	GAS	71	6	19	26.76	13	18.31
	LIGHT LIQUID	54	5	17	31.48	9	16.67
	HEAVY LIQUID	3	0	1	33.33	0	0.00
RELIEF VALVES	GAS	0	67	0	0.00	0	0.00
	LIGHT LIQUID	0	14	0	0.00	0	0.00
VALVES	GAS	1970	455	596	30.36	395	20.05
	LIGHT LIQUID	1990	305	714	35.88	482	24.22
	HEAVY LIQUID	54	0	5	9.26	2	3.70
PUMPS	LIGHT LIQUID	35	0	14	40.00	10	28.57
COMPRESSORS*	GAS	3	0	0	0.00	0	0.00

* The screening was conducted at the flange separating the compressor housing from the drive shaft shroud.

Unit 2

1. PROCESS DESCRIPTION

Process unit number two is an olefins manufacturing unit with the primary products being ethylene, propylene, butylenes, butadiene, and naphtha. The manufacturing process is shown in Figure 3. The primary products are manufactured by pyrolysis of gas phase (ethane/propane) and liquid phase (butane/naphtha) feedstocks. The reaction products from the cracking furnaces are separated in an extensive system of refrigerated distillation columns. A butadiene extraction system and a naphtha hydrotreating system are physically integrated with the olefins unit, but they were excluded from the screening effort as downstream processing.

Every attempt was made to trim the unit definition to minimize the screening effort. Where identical parallel processing trains were used, only the smallest complete system was screened. For instance, only two of the eight cracking furnaces were screened, one charging ethane/propane as a vapor and one charging butane as a liquid. Naphtha was not charged to the unit during the survey. All of the quench, compression, refrigeration, and separation systems were screened through the debutanizer column.

2. SAMPLING PROBLEMS AND OBSERVATIONS

Almost 4,500 valves were identified, but 800 were not screened. Almost half of these unscreened valves were in elevated locations that could have been accessed only by using extensive scaffolding, a crane, or a "cherry picker". About 350 unscreened valves could have been reached by using a ladder. Miscellaneous conditions, such as heavy ice formation, blowing steam, and obstructions from adjacent piping, prevented screening of 70 valves.

Many process valves in cold service were partially covered with ice. Fugitive emissions from these valves were often difficult to detect, because emissions escaped from underneath the ice cap at random locations. Accurate

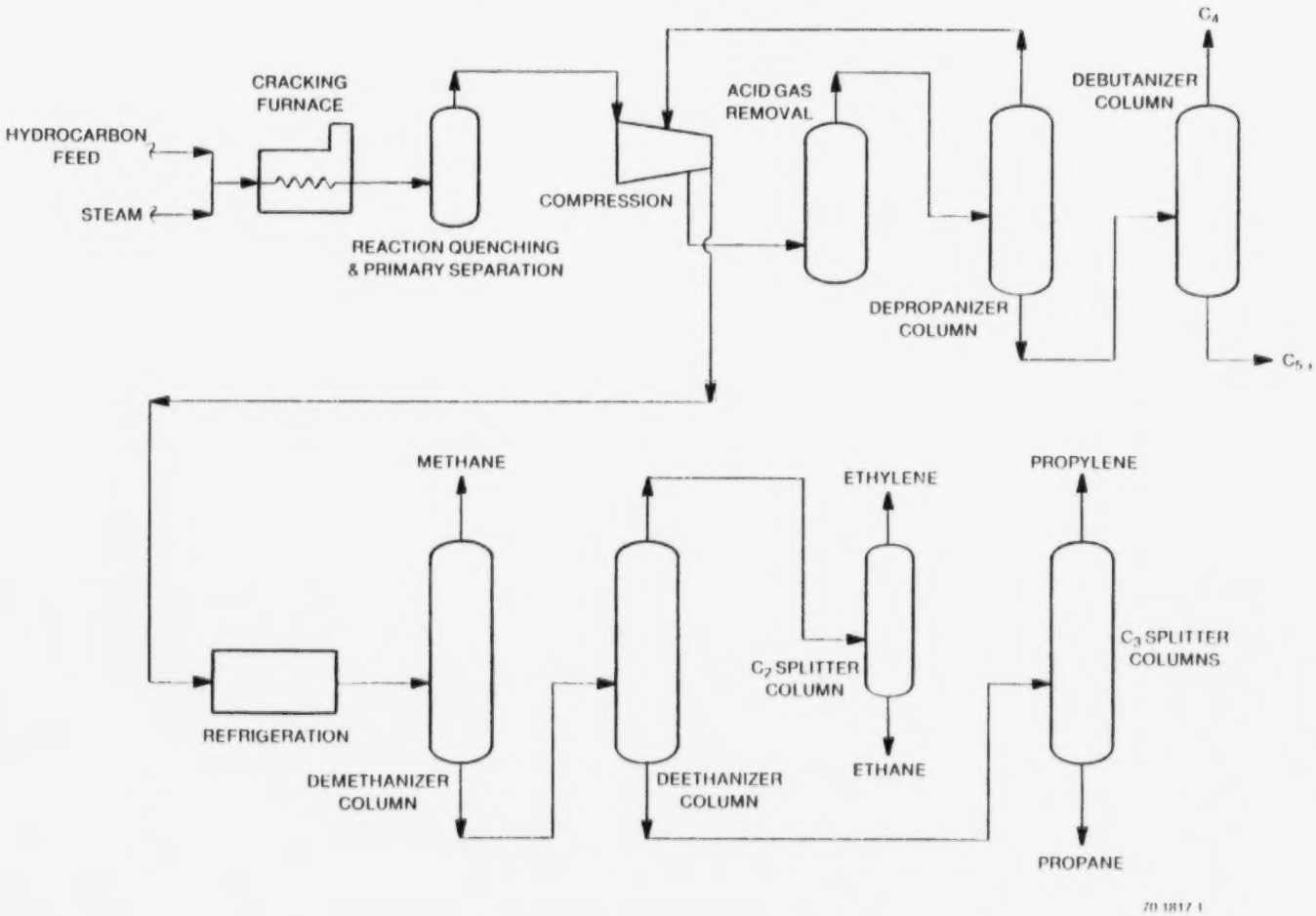


FIGURE 3 PROCESS FLOW DIAGRAM - UNIT 2 (ETHYLENE)

screening values from these ice-covered valves were difficult to obtain; care had to be taken to prevent moisture on wet valve surfaces from being drawn into the instrument probe.

The relief valves in process unit number two were vented to a flare header. Therefore, these relief valves were not screened, although they were physically accessible.

Twenty-four pumps with single mechanical seals and three pumps with double mechanical seals were screened at the seal area. The remaining 11 pumps were not screened because drive shaft guards or ice concealed the seal area. Four of these pumps had single mechanical seals; six were equipped with double mechanical seals. One of these pumps had a single packed seal.

The three compressors in this process unit were equipped with labyrinth seal systems, which were enclosed and vented to the flare. These compressors were screened at the flange separating the compressor housing from the shroud enclosing the drive shaft.

3. SAMPLING TIME

The screening of process unit number two required 13 working days between February 12 and February 28, 1980. One two-person team was in operation for that entire period, and they were joined by a second team for six working days.

Almost 6,000 potential fugitive emission sources were identified, although only 5,100 sources were actually screened. Source screening, including instrument calibration, required 110 total hours (220 person-hours). Therefore, screening of sources was conducted at an average rate of 1.3 minutes per source for a two-person team (or about 2.6 person-minutes per source).

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT #2

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	142	0	17	11.97	5	3.52
	LIGHT LIQUID	51	0	3	5.88	1	1.96
	HEAVY LIQUID	73	1	0	0.00	0	0.00
PROCESS DRAINS	GAS	2	0	1	50.00	1	50.00
	LIGHT LIQUID	94	6	1	1.06	0	0.00
	HEAVY LIQUID	14	0	1	7.14	1	7.14
OPEN ENDED LINES	GAS	50	6	13	26.00	11	22.00
	LIGHT LIQUID	38	1	15	39.47	12	31.58
	HEAVY LIQUID	89	5	0	0.00	0	0.00
RELIEF VALVES	GAS	0	42	0	0.00	0	0.00
	LIGHT LIQUID	0	4	0	0.00	0	0.00
	HEAVY LIQUID	0	1	0	0.00	0	0.00
VALVES	GAS	2153	410	437	20.30	318	14.77
	LIGHT LIQUID	1191	221	395	33.17	309	25.94
	HEAVY LIQUID	1144	167	18	1.57	11	0.96
PUMPS	LIGHT LIQUID	16	6	9	56.25	6	37.50
	HEAVY LIQUID	15	1	0	0.00	0	0.00
COMPRESSORS	GAS	7	1	1	14.29	1	14.29

APPENDIX H -- METHYL ETHYL KETONE

UNIT 31

SECTION 1

PROCESS DESCRIPTION

Methyl ethyl ketone (MEK) is manufactured at this facility by the catalytic dehydrogenation of sec-butyl alcohol(SBA). The reaction equation is:

$$2CH_3\overset{OH}{\overset{|}{C}}HCH_2CH_3 \xrightarrow{\text{catalyst}} 2CH_3\overset{O}{\overset{\|}{C}}CH_2CH_3 + H_2$$

A simplified flow diagram of the process used at this facility is shown in Figure 1. As can be seen, an SBA feed stream enters the process from tankage. This stream is used to absorb MEK and SBA from the hydrogen steam produced in the dehydrogenation reaction. This SBA stream is then heated and fed into the dehydrogenation reactor section.

In the reactor section, the feed SBA stream is heated and reacted over a zinc or zinc oxide based catalyst to form crude MEK. The reaction takes place at a temperature of about 800^{o}F (425^{o}C). The product stream from the reactors is heat exchanged with the feed stream and then flashed to separate hydrogen from the crude MEK stream. The crude MEK stream enters the purification section of the process.

The crude MEK stream is stripped of hydrogren in the first operation in this section. The product stream from this operation is further fractionated to produce a product MEK stream. The unreacted SBA recovered in this section is recycled to the process.

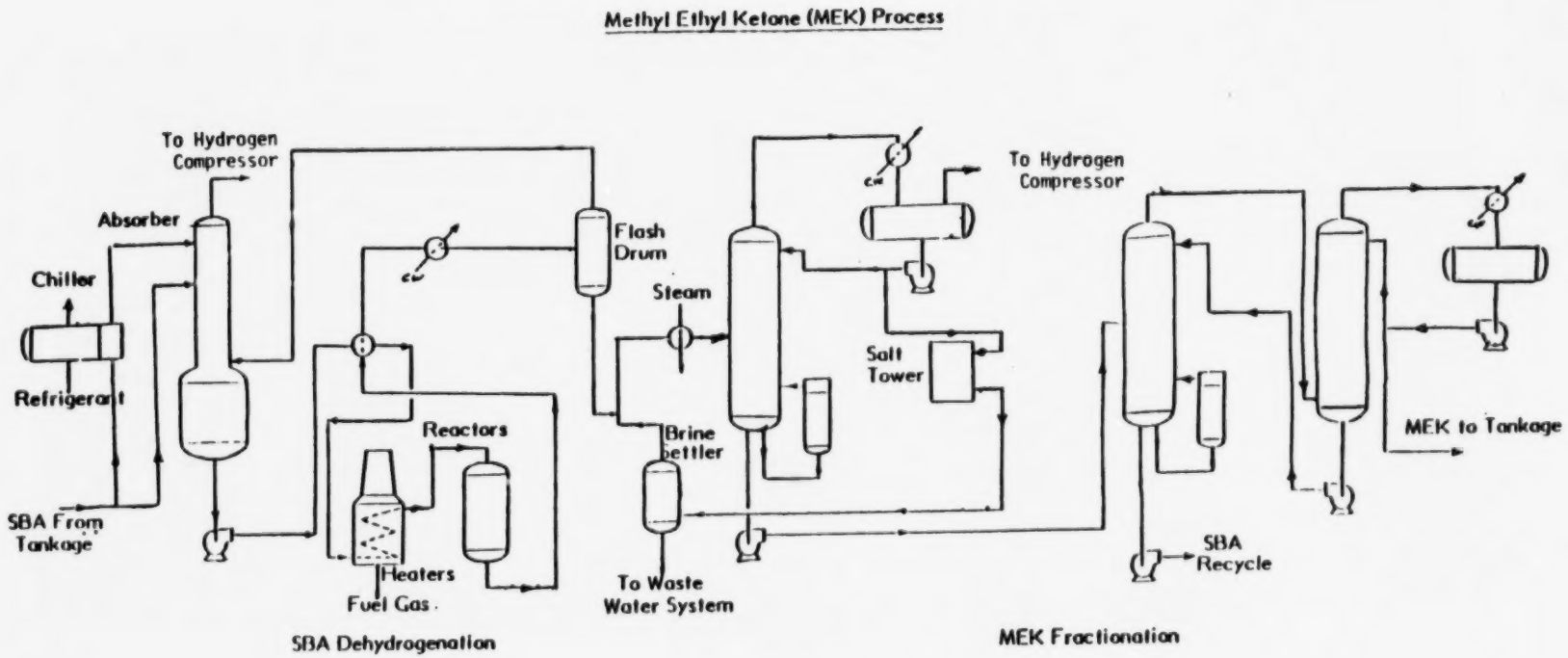


Figure 1. Methyl ethyl ketone unit.

SECTION 2

SAMPLING PROBLEMS & OBSERVATIONS

Sampling at this MEK facility proceeded in three steps. The steps are:

- MEK unit boundaries were established
- Process information was gathered
- Process sources were screened

These steps are dicussed below.

2.1 MEK UNIT BOUNDARIES

A report[1] was furnished to Acurex by the EPA which included a general process description of the MEK process to be sampled. Based on this report and discussions with plant personnel, it was decided that the MEK unit boundary began at the feed of "product grade" SBA to the conversion section and ended with the exit of "product grade" MEK to tankage. This definition excluded those sections that are involved in the conversion of mixed C_4's to SBA.

2.2 PROCESS INFORMATION

The process information had been gathered by plant personnel before the Acurex sampling crew arrived onsite. This data was checked at intervals against pressure and temperature indicators in the unit. No discrepancy was seen between the data furnished by plant personnel and that observed in the field.

2.3 PROCESS SOURCES

The process sources screened were:

- Valves
- Pump seals
- Relief valves
- Process drains
- Flanges
- Open-ended lines

All accessible sources in the above categories except for flanges, were screened. Approximately five percent of the flange population were screened. All screened sources were numbered according to their corresponding number on the data sheets. Sources were not screened if they could not be reached from a permanent ladder, stairway, or platform.

The screening technique used followed that specified in an EPA approved sampling plan. This sampling plan was reviewed and approved by the Acurex statistician.

No problems were encountered in the sampling activity. Problems were encountered in securing the correct calibration gases though. These problems were corrected and sampling proceeded smoothly after several days delay.

SECTION 3

SAMPLING TIME

Sampling at this process unit required a total of 32 man-hours to complete. The breakdown of the man-hours expended is:

Activity	Time, Man-hours
Instrument calibration and sampling preparation	6
Source sampling	26

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT=51

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	6	0	0	0.00	0	0.00
	LIGHT LIQUID	35	0	2	5.71	0	0.00
PROCESS DRAINS	GAS	6	0	4	66.67	1	16.67
	LIGHT LIQUID	14	3	5	35.71	3	21.43
OPEN ENDED LINES	GAS	12	0	4	33.33	1	8.33
	LIGHT LIQUID	93	14	25	26.88	8	8.60
AGITATOR SEALS	GAS	1	0	0	0.00	0	0.00
RELIEF VALVES	GAS	1	0	0	0.00	0	0.00
VALVES	GAS	81	1	28	34.57	15	18.52
	LIGHT LIQUID	321	27	45	14.02	17	5.30
PUMPS	LIGHT LIQUID	15	0	4	26.67	0	0.00

UNIT 32

SECTION 1

PROCESS DESCRIPTION

Secondary Butyl Alcohol (SBA) is converted to Methyl Ethyl Ketone (MEK) in two major processing steps. In the first step, the alcohol is passed over a dehydrogenation catalyst at high temperatures. The conversion is partially complete, yielding a crude product containing MEK, by-product hydrogen, light and heavy impurities, and unconverted SBA. The hydrogen is scrubbed and utilized in the fuel system.

In the second step, the crude MEK is distilled into pure MEK, light impurities, heavy impurities, and unconverted SBA streams. The unreacted SBA is recycled back to the reactor section. The reaction equation for this process is:

$$2\ CH_3\ \underset{|}{\overset{OH}{CH}}\ CH_2\ CH_3 \xrightarrow{\text{catalyst}} 2\ CH_3\ \overset{O}{\overset{||}{C}}\ CH_2\ CH_3 + H_2$$

A simplified process flow plan for the MEK plant is shown in Figure 2 As can be seen, finished SBA is fed to an alcohol scrubber (1) where MEK is scrubbed from the section's effluent hydrogen stream. The alcohol, now containing some MEK, is preheated by reactor effluent (2) prior to being vaporized and superheated in the furnaces (3). The dehydrogenation reaction takes place in the vapor phase over a solid catalyst. The reactor effluent is cooled (4) to separate MEK and unconverted SBA from the by-product hydrogen. The crude MEK is pumped to intermediate tankage. The hydrogen is

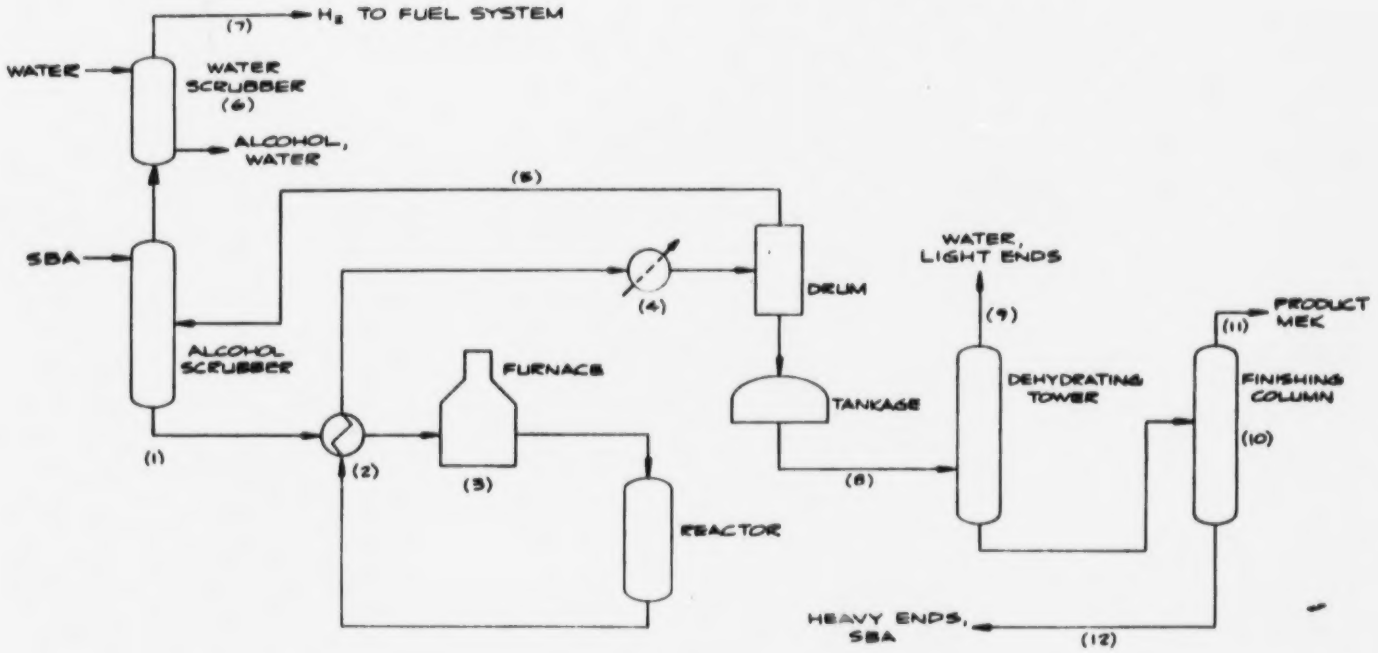


Figure 2. Simplified Process Flow Diagram of the MEK Unit Sampled

scrubbed (5) with incoming fresh feed to recover the MEK, then scrubbed with water to recover the alcohol from that stream (6). The hydrogen/water stream is used as the primary fuel for this unit's furnaces (7).

MEK finishing occurs in two distillation columns. Crude MEK produced in the conversion section is fed (8) to a dehydrating tower where light ends and water are removed from the system as an overhead stream (9). The bottoms of this column are then fed to a finishing column (10) where the product grade MEK is taken overhead (11). Heavy ends and unconverted SBA (12) are recycled and recovered.

SECTION 2

SAMPLING PROBLEMS AND OBSERVATIONS

Sampling at this MEK facility proceeded in three steps. These three steps are:

- Establish MEK unit boundaries
- Screen process sources
- Obtain process information

These three steps are discussed below.

2.1 MEK UNIT BOUNDARIES

The MEK unit boundaries were set according to the definition of an MEK unit presented in a report furnished Acurex by the EPA[1]. The unit boundaries were established to include the MEK conversion and finishing sections. The conversion section begins at the inlet of "product grade" SBA and the finishing section ends where "product grade" MEK exits to tankage. This unit definition is consistent with the unit definition used in sampling another MEK unit.

2.2 PROCESS SOURCES

Process sources screened at this MEK facility include:

- Valves
- Pump seals
- Compressor seals
- Process drains

- Relief vlaves
- Flanges
- Open ended lines

All accessible sources in these groups were screened, except for flanges. Approximately five percent of the total flange population were screened. All screened sources were numbered according to the corresponding number on the data sheet. The number and location of each source was recorded for use in completing the data sheets. Sources were not screened if they could not be reached from a permanent ladder, stairway, or platform.

Acurex's sampling procedures corresponded to those presented in a detailed sampling plan furnished by the EPA.[2]. No problems were encountered in the sampling activities.

2.3 PROCESS INFORMATION

The process information was assembled once screening had been completed. This information was assembled from mass balances around key process equipment. All pressure and temperature data taken from the mass balances were within the prescribed ranges when checked against indicators in the process unit.

SECTION 3

SAMPLING TIME

The sampling at this process unit required a total of 50 man-hours to complete. The breakdown of time expended is:

Activity	Time, Man-hours
Instrument calibration and sampling preparation	8
Source sampling	42

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT=32

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	16	0	0	0.00	0	0.00
	LIGHT LIQUID	41	0	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	9	0	4	44.44	2	22.22
OPEN ENDED LINES	GAS	25	2	3	12.00	2	8.00
	LIGHT LIQUID	93	4	26	27.96	11	11.83
RELIEF VALVES	GAS	2	0	0	0.00	0	0.00
VALVES	GAS	126	43	9	7.14	4	3.17
	LIGHT LIQUID	350	59	53	15.14	17	4.86
PUMPS	LIGHT LIQUID	16	0	4	25.00	1	6.25
COMPRESSORS	GAS	1	0	1	100.00	1	100.00

REFERENCES

1. Key, J. A., "Emissions Control Options for the Synthetic Organic Chemicals Manufacturing Industry," Hydroscience Draft Report, EPA Contract 68-02-2577, Hydroscience, Incorporated, Knoxville, Tennessee, March 1979.

2. Harris, G. E. and Langley, G. J., "Detailed Screening Plan for Fugitive Emissions from SOCMI Process Units," Radian Corporation, Austin, Texas, December 1979.

APPENDIX I

FORMALDEHYDE

PROCESS DESCRIPTION

Plant 22 uses the silver catalyst process in production of formaldehyde. The process has three steps: (1) methanol dehydration, (2) formaldehyde absorption, and (3) formaldehyde purification. Figure 1 illustrates the basic process flow at the facility.

Methanol Dehydration

The process starts with the vaporization of methanol with steam. Air is washed with caustic to remove impurities such as sulfur dioxide and hydrogen sulfide. The washed air and methanol react in a converter at elevated temperatures in the presence of a silver catalyst. The following dehydration reaction occurs:

$$\underset{\text{(methanol)}}{CH_3OH} \rightarrow \underset{\text{(formaldehyde)}}{HCHO} + \underset{\text{(hydrogen)}}{H_2}$$

A portion of the hydrogen is oxidized to water. The silver catalyst process maintains an excess of methanol and operates above the explosive range.

Formaldehyde Absorption

The reaction gases are cooled to prevent decomposition of the formaldehyde formed. The primary absorber is an aqueous solution of formaldehyde and methanol. A portion of the absorber liquid is pumped to a fractionation column for purification. The uncondensed vapors and noncondensable gases from the primary absorber are fed to a secondary absorber, where the uncondensed vapors are recovered as demineralized water. The resultant weak solution is used as makeup to the primary absorber. Off-gases from the secondary adsorber are vented to the atmosphere, processed, or incinerated.

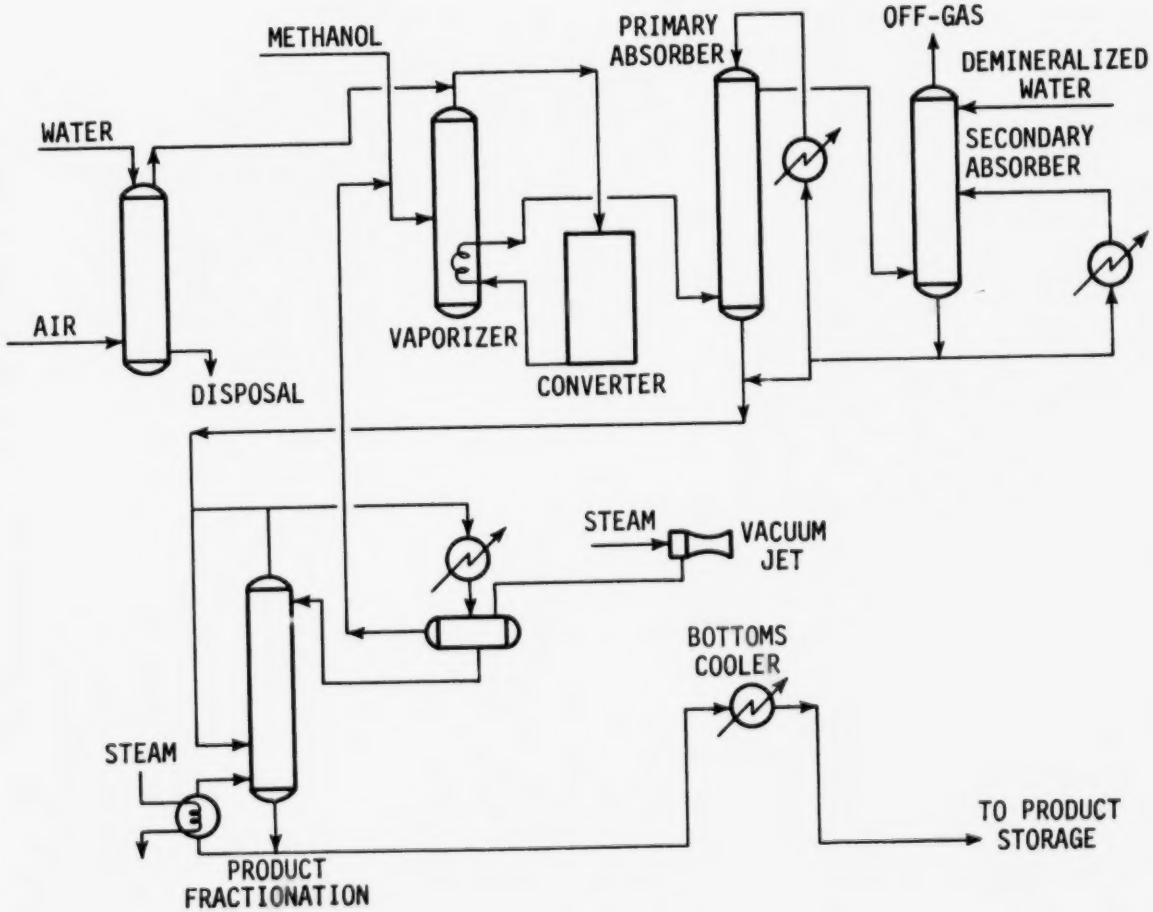


Figure 1. Formaldehyde from methanol using the metallic-silver-catalyst process.

Formaldehyde Purification

A portion of the methanol/formaldehyde solution undergoes purification in the fractionation column. The overhead product is primarily methanol which is condensed and recycled to the reactor. Uncondensed vapors are vented. The bottoms from the fractionator column is formalin. The formaldehyde is pumped to product storage tanks, where demineralized water is added to meet customer specifications.

SAMPLING PROBLEMS AND OBSERVATIONS

The instruments used by PEDCo in the survey were the flame-ionization hydrocarbon detector produced by Century Systems and the photoionization hydrocarbon detector produced by HNU Systems, Inc. The Century Systems Organic Vapor Analyzer Model OVA-128 was used with the dilution probe. The OVA-128 has a linear readout with a range from 1 to 1000 ppmv. Use of the dilution probe on the OVA-128 provides capabilities of up to 10,000 ppmv with 10 to 1 dilution of the sample stream. The HNU P1-101 has a linear readout with a range from 1 to 2000 ppmv. For use in this study, the instruments were as follows:

Code	
2	OVA-128
3	NIOSH HNU with 11.7-μv probe, calibrated on March 3, 1980. It was never used to screen in the field. On March 5, 1980, it was used with the 10.2-μv probe.
4	EPA HNU with 11.7-μv probe.

Among the quality control procedures followed to ensure reliability of the data, the most important was daily calibration of the hydrocarbon detectors. In addition to the morning calibration recommended by EPA, the PEDCo teams conducted an evening check to determine any calibration drift that may have occurred. The technique used for calibrating the OVA-108 was established by EPA; the procedure for calibrating the OVA-128 was modified to accommodate the dilution probe. Adjustments to the high calibration standard with the dilution probe attached were made with the pinch clamp regulating the airflow

through the diluter. The gas select knob adjusted the low calibration standard without the dilution probe, with the instrument operating in the 100X mode. The low and high calibration standards consisted of methane gas at concentrations of 491 and 8450 ppm. The standard for calibrating the HNU was isobutylene at 451 ppm in air. The instrument was calibrated in the morning and rechecked in the evening. As a crosscheck, the isobutylene concentration was measured with the OVA, calibrated on methane. The repeat screening procedure outlined in the EPA manual was followed, with three methanol sources. Both the OVA-128 and the HNU read methanol. Only two leaks occurred with high concentrations of formaldehyde. Since the OVA-128 did not read these sources, repeat screening would have been futile. The repeat screening values varied from day to day.

Since the process unit was relatively compact, the screening procedure followed the process flow. We did not tag the screened sources because the unit was small and the process flow could be followed easily. Plant personnel prepared a sketch of each screened unit as an aid to locating the repeat screening sources and preventing duplication. The formaldehyde plant is a single unit. For screening purposes, it was designated code "22."

The screening team completed the material coding sheet before entering the plant. Because methanol and formaldehyde were the only two volatile chemicals involved, we could determine the product concentrations relatively easily. An engineer familiar with the process indicated product concentrations as we screened the unit.

No problems occurred with the diluter pinch clamp during the two times it was used. We had no problems with Tygon tubing as we did in the plants producing EDC. The OVA-128 was used at this plant because the OVA-108 was being repaired. Both instruments had been used the previous week in screening of petroleum refineries. It appears that constant use of this equipment necessitates weekly maintenance of the ignitor and combustion chamber because heavy organics and chlorinated solvents cause these components to deteriorate.

A problem with the OVA-128 led to flameouts and improper scale response. The instrument scale selector malfunctioned during the screening, and the instrument had to be dismantled for repair. In discussion by telephone, sales representatives of Digicolor in Columbus diagnosed the problem as faulty contacts in the combustor thermocouple. We restored proper operation by

tightening the contacts. Digicolor recommended shorting the thermocouple from the circuit with a paper clip if the problem recurred, but it did not.

Problems also developed with the HNU instrument, whose probe cable is much stiffer than that of the OVA. The NIOSH HNU had been used much more heavily than the EPA HNU, and heavy use stresses the coupling between the probe and instrument. The NIOSH HNU shorted out occasionally as the probe was manipulated during calibration. This problem did not occur with the EPA HNU, which was used during the screening.

Both the HNU and the OVA read background hydrocarbon levels (10 to 20 ppm normally). In the event of a short (HNU) or a flameout (OVA), the instrument reading drops to zero. In values shown on the data sheets, the background level has been subtracted from the instrument reading.

The readout of the HNU is in the instrument case rather than on the probe, like the OVA readout. This placement causes some inconvenience in screening and requires good eye and hand coordination to obtain an accurate reading. Operators often moved the probe from the source while concentrating on the indicator.

Lack of a specific calibrating gas is a problem with detection of formaldehyde. Isobutylene in air was used to calibrate the HNU because formaldehyde standards are unstable.

During the calibration period, the HNU with the 10.2-μv bulb recorded no high concentrations of formaldehyde or methanol. The bulb apparently lacked the energy needed to activate the electrons that would detect a leak; therefore the 11.7-μv bulb was used. For the two leaks with high formaldehyde concentrations, the OVA did not indicate a leak but the HNU with the 11.7 μv bulb did.

Another problem was formaldehyde solidification. We tested a number of sources that had solid residue, which indicated leaks, but no VOC emissions were measured. Because formaldehyde polymerizes to form solid paraformaldehyde, which is impervious to formaldehyde vapor, the formaldehyde leaks tend to be self-sealing.

Only a relatively few flanges were screened during this study because the plant was quite small. The screening team tried to screen one of every twenty flanges. Many sources were sample and instrument taps of the main fractionating column, which reduced the percentage of flanges screened.

SAMPLING TIME

The formaldehyde plant was relatively compact. One team of three persons (one logger and two instrument operators) screened for 6 hours the first day. The second day a three-person team screened for about 3 hours. The screening entailed a total of 27 labor-hours.

4. DATA SUMMARY

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SUMMARY STATISTICS
FORMALDEHYDE
UNIT 22 - OVA

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	2	0	0	0.00	0	0.00
	LIGHT LIQUID	8	0	2	25.00	1	12.50
OPEN ENDED LINES	GAS	14	2	0	0.00	0	0.00
	LIGHT LIQUID	36	1	1	2.78	0	0.00
RELIEF VALVES	GAS	0	2	0	0.00	0	0.00
	LIGHT LIQUID	0	1	0	0.00	0	0.00
VALVES	GAS	41	7	1	2.44	1	2.44
	LIGHT LIQUID	121	5	1	0.83	0	0.00
PUMPS	LIGHT LIQUID	8	0	0	0.00	0	0.00

4. DATA SUMMARY

****

SUMMARY STATISTICS
FORMALDEHYDE
UNIT 22 - HNU

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	2	0	0	0.00	0	0.00
	LIGHT LIQUID	8	0	0	0.00	0	0.00
OPEN ENDED LINES	GAS	14	2	0	0.00	0	0.00
	LIGHT LIQUID	36	1	1	2.78	0	0.00
RELIEF VALVES	GAS	0	2	0	0.00	0	0.00
	LIGHT LIQUID	0	1	0	0.00	0	0.00
VALVES	GAS	41	7	0	0.00	0	0.00
	LIGHT LIQUID	121	5	0	0.00	0	0.00
PUMPS	LIGHT LIQUID	8	0	0	0.00	0	0.00

APPENDIX J -- METHYL METHACRYLATE

UNIT 34

SECTION 1

PROCESS DESCRIPTION

Methyl methacrylate is produced via a two-stage reaction. In the first stage, acetone cyanohydrin and sulfuric acid are reacted to produce methacrylamide hydrogen sulfate by the following reaction:

$$(CH_3)_2COCHCN + H_2SO_4 \longrightarrow (CH_2 = CCH_3CONH_3)HSO_4 \qquad (2\text{-}1)$$

acetone cyanohydrin — sulfuric acid — methacrylamide hydrogen sulface

The intermediate methacrylamide hydrogen sulfate is then esterified with methyl alcohol to produce methyl methacrylate. The reaction is as follows:

$$(CH_2 = CCH_3CONH_3)HSO_4 + CH_3OH \longrightarrow CH_2 = CCH_3COOCH_3 + NH_4HSO_4 \qquad (2\text{-}2)$$

methacrylamide hydrogen sulfate — methyl alcohol — methyl methacrylate ammonium bisulfate

A simplified flow diagram of the methyl methacrylate process is shown in Figure 1. In the first step in the process, acetone cyanohydrin and concentrated sulfuric acid are reacted in a hydrolysis kettle to form the intermediate methacrylamide hydrogen sulfate. The hydrolysis reactor effluent is sent to an esterification reactor to react continuously with methyl alcohol. The effluent from the esterification reactor is sent to a separator where methyl alcohol and methacrylic acid are recovered and recycled to a

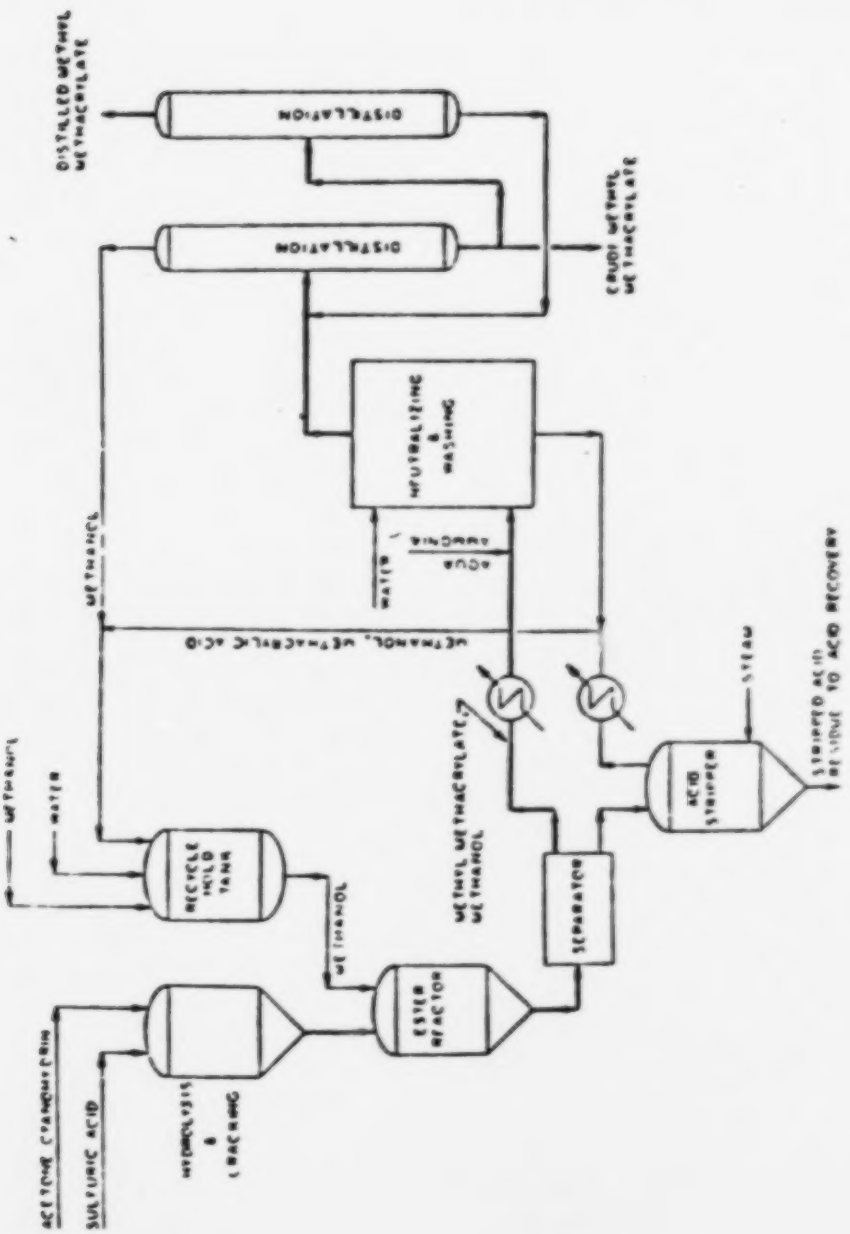

Figure 1. A Simplified Process Flow Diagram for the Methyl Methacrylate Unit Sampled

holding tank. The bottoms of the acid stripper are sent to the acid recovery unit. The organic layer from the separator is neutralized by aqueous ammonia, and washed with water in a washing column.

Methanol is recovered from the overhead of the washing column by distillation, and is condensed and recycled to the recycle hold tank. The bottoms stream from the distillation column is technical-grade methyl methacrylate. Distilled methyl methacrylate product is produced from technical grade methyl methacrylate in another distillation column.

SECTION 2

SAMPLING PROBLEMS & OBSERVATIONS

Sampling at this facility proceeded in three steps. These three steps are:

- Establish unit boundaries
- Screen process sources
- Obtain process information

These steps are discussed below.

2.1 UNIT BOUNDARIES

The unit boundaries set for the purpose of this study are based on the process unit defined in the previous section of this appendix. This definition was provided by the host company. These unit boundaries correspond to the battery limits for the process unit.

2.2 PROCESS SOURCES

Process sources screened at this facility include:

- Valves
- Pump seals
- Agitator seals
- Process drains
- Relief valves
- Flanges
- Open ended lines

All accessible sources in these groups were screened, except for flanges, of which approximately 20 percent of the total population were screened. Approximately 30 percent of the screened flanges were covered. On these occasions, the sources were screened at the edge of the cover. All screened sources were numbered according to the corresponding number on the data sheet. Sources were not screened if they could not be reached from a permanent ladder, stairway, or platform. Most of the sources not screened at this facility would have required scaffolding or walking out on pipe racks to sample.

Acurex's sampling procedures correspond to those presented in a detailed sampling plan furnished by the EPA. No problems in either instrumentation or procedure were encountered in this sampling activity.

2.3 PROCESS INFORMATION

The process information was assembled by the unit process engineer for each day's sampling. At the host company's request, the accuracy ranges on process line temperatures and process line pressures on the data sheets were expanded from $\pm 25^{o}F$ and ± 25 psig, respectively, to $\pm 50^{o}F$ and ± 50 psig, respectively. The process information given by the host company was within the expanded ranges when checked against pressure and temperature indicators in the process unit.

SECTION3

SAMPLING TIME

The sampling activity at this facility required a total of 62.5 man-hours to complete. The breakdown of time spent is shown below.

Activity	Time, Man-hours
Instrument activity calibration and sampling preparation	10
Source sampling	52.5

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT #34

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	38	0	0	0.00	0	0.00
	LIGHT LIQUID	247	0	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	21	0	0	0.00	0	0.00
OPEN ENDED LINES	GAS	63	10	0	0.00	0	0.00
	LIGHT LIQUID	335	16	10	2.99	1	0.30
AGITATOR SEALS	GAS	3	0	2	66.67	1	33.33
	LIGHT LIQUID	3	1	0	0.00	0	0.00
RELIEF VALVES	GAS	4	2	0	0.00	0	0.00
	LIGHT LIQUID	12	3	1	8.33	1	8.33
VALVES	GAS	190	30	0	0.00	0	0.00
	LIGHT LIQUID	1058	121	2	0.19	1	0.09
PUMPS	LIGHT LIQUID	45	4	4	8.89	2	4.44

APPENDIX K - Vinyl Acetate

Unit 1

1. PROCESS DESCRIPTION

Process unit number one is a vinyl acetate manufacturing facility. The manufacturing process is shown in Figure 1. Ethylene, oxygen, and acetic acid are catalytically reacted to form the crude vinyl acetate. A series of fractionating columns is used to remove unreacted feed materials for recycle and by-products such as acetaldehyde, methyl acetate, and ethyl acetate. Hydroquinone is added at several points in the refining section as a polymerization inhibitor.

The entire unit, as defined by a single continuous pad, was screened. This included the reaction section, the refining section, and a building used for preparing the hydroquinone solution.

2. SAMPLING PROBLEMS AND OBSERVATIONS

Approximately 1,130 process valves were identified in process unit number one. Of these, 100 valves were not screened. Two-thirds of these unscreened valves could have been reached by using a ladder. The remaining 30 valves could only have been reached with the use of extensive scaffolding, a crane, or a "cherry picker."

About two-thirds of the 49 pumps in this vinyl acetate unit were equipped with double mechanical seals. The remaining pumps had single mechanical or packed seals. All pumps were screened at the seals.

Three single mechanical seals in compressor service were screened. One compressor's double mechanical seal area was enclosed, and the seal oil system was vented to a flare. This system was screened at a vacuum breaker on the seal oil reservoir.

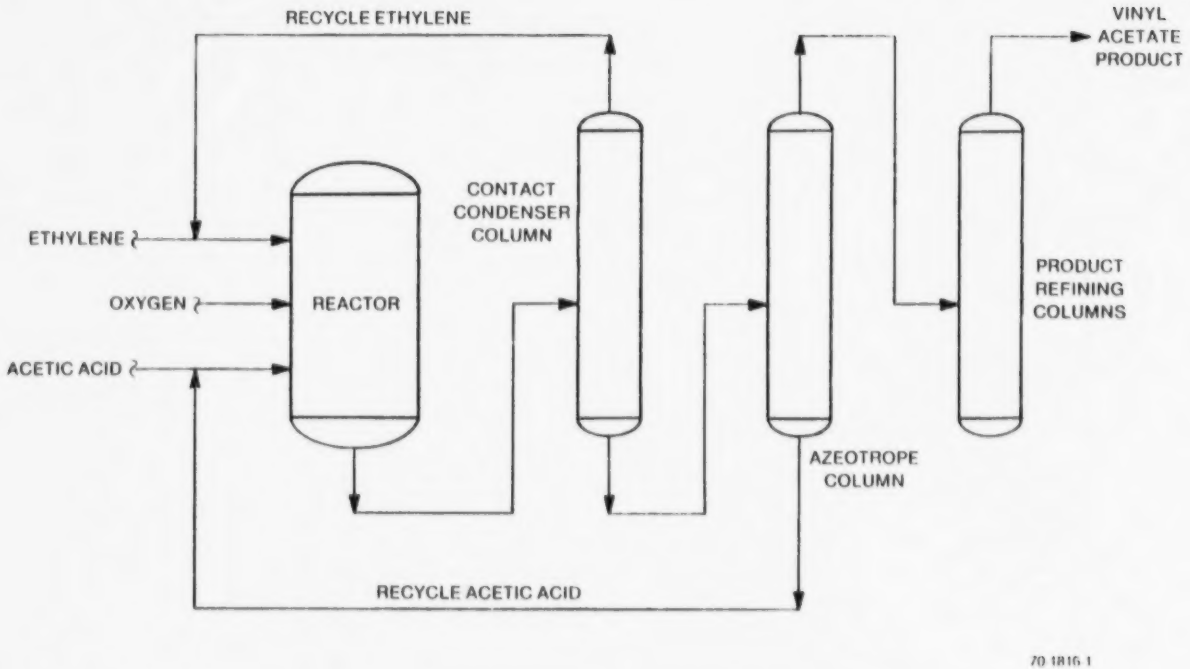

FIGURE 1 PROCESS FLOW DIAGRAM - UNIT 1 (VINYL ACETATE)

Most of the 21 relief valves in process unit number one were connected directly to a flare header. Therefore, these relief valves' vents could not be screened. Two relief valves were not vented to the flare, and they were screened.

Problems developed with the primary screening instrument and its backup because of rainy weather. Moisture was inadvertently drawn into the instruments, resulting in false readings. Allowing the instruments to run (and dry out) overnight corrected the problem. Because a backup instrument was available, no time was lost from the screening effort.

3. SAMPLING TIME

Screening of process unit number one required six working days from January 22 to January 29, 1980. A two-person team screened for this entire period, and they were joined by another two-person team for two working days. Including time spent for instrument calibration, screening required a total of 47 hours (94 person-hours).

Approximately 1,520 sources were identified, of which 1,390 were actually screened. The initial screening rate was low, from 30 to 90 screened sources per team per day, because of poor weather and lack of familiarity with the screening procedures. On the average, screening was conducted at a rate of 2.0 minutes per source for a two-person team (or about 4.0 person-minutes per source).

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT=1

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	38	0	1	2.63	1	2.63
	LIGHT LIQUID	56	0	0	0.00	0	0.00
	HEAVY LIQUID	3	0	0	0.00	0	0.00
OPEN ENDED LINES	GAS	37	0	1	2.70	1	2.70
	LIGHT LIQUID	46	3	2	4.35	0	0.00
	HEAVY LIQUID	8	0	0	0.00	0	0.00
AGITATOR SEALS	LIGHT LIQUID	3	0	0	0.00	0	0.00
	HEAVY LIQUID	1	0	0	0.00	0	0.00
RELIEF VALVES	GAS	0	15	0	0.00	0	0.00
	LIGHT LIQUID	2	4	0	0.00	0	0.00
VALVES	GAS	529	48	19	5.78	12	3.65
	LIGHT LIQUID	736	52	6	0.82	1	0.14
	HEAVY LIQUID	67	0	0	0.00	0	0.00
PUMPS	LIGHT LIQUID	44	0	5	11.36	3	6.82
	HEAVY LIQUID	5	0	0	0.00	0	0.00
COMPRESSORS	GAS	4	0	0	0.00	0	0.00
OTHER[1]	GAS	6	0	1	16.67	1	16.67
	LIGHT LIQUID	6	1	0	0.00	0	0.00

[1]Includes filter and vacuum breaker.

Unit 3

1. PROCESS DESCRIPTION

Process unit number three is a vinyl acetate manufacturing facility. The manufacturing process is shown in Figure 2. Ethylene, oxygen, and acetic acid are catalytically reacted to form the crude vinyl acetate product. A series of fractionating columns removes unreacted feed materials for recycle and by-products such as acetaldehyde and ethyl acetate.

The screening effort was focused on four systems within the vinyl acetate process unit. These were the reaction, distillation, polymer removal, and CO_2 removal systems. These systems included the delivery of make-up and recycle acetic acid and ethylene to the unit up through delivery of the refined vinyl acetate product from the unit. Also screened were polymerization inhibitor lines, seal liquid systems, and the ethylene blowdown and compressor buffer gas system. The waste incinerator, flare, vessel blowdown tower, lubricating oil systems, and storage facilities were not screened.

2. SAMPLING PROBLEMS AND OBSERVATIONS

Over 2,500 process valves were identified, but only 2,080 were screened. About two-thirds of the 430 unscreened valves could have been reached with a ladder. About five percent of the unscreened valves were in locations that were unsafe for screening. These locations included the area in which oxygen was injected into the reactor feed and difficult-to-access piping around a compressor installation. The remaining unscreened valves were at elevated locations that could have been accessed only with extensive scaffolding, a crane, or a "cherry picker".

Only 12 of the 49 pumps in process unit number three were screened directly at the seal. Safety guards made from metal sheets were installed to cover the shafts of the remaining pumps and prevented direct access to the seal area. For these pumps, screening values

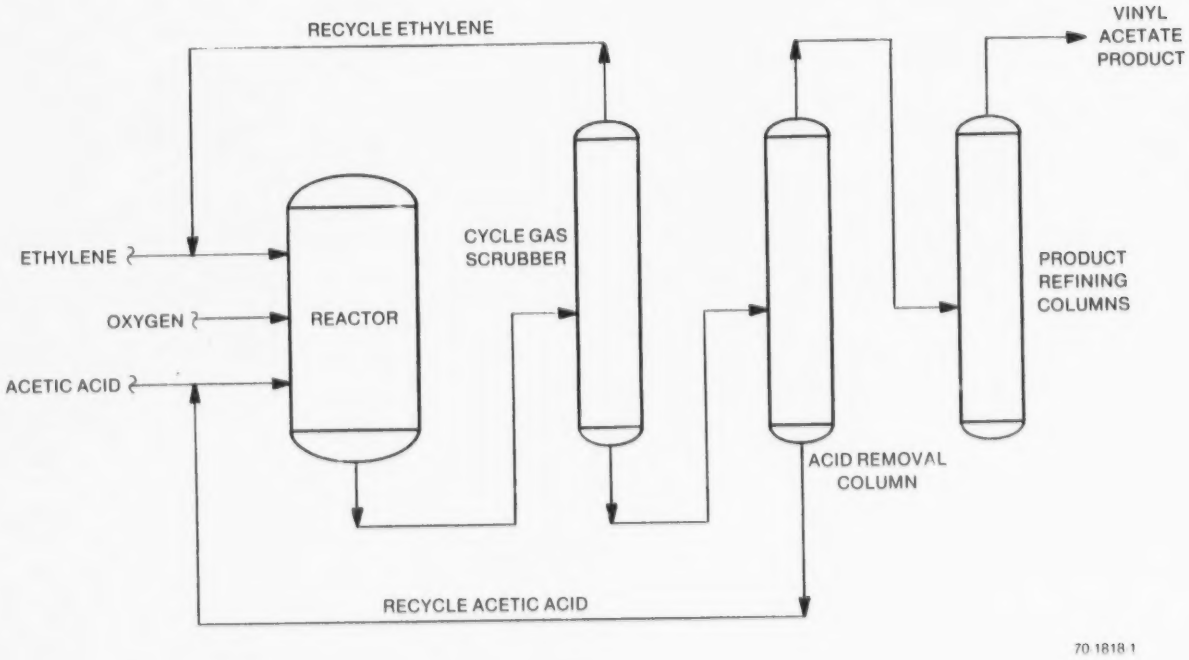

FIGURE 2 PROCESS FLOW DIAGRAM - UNIT 3 (VINYL ACETATE)

were obtained with the instrument probe held as near as possible to the seal. All pumps screened had single mechanical seals.

The two compressors in the process unit were equipped with double mechanical seals. The compressor drive shafts were open to the atmosphere but they were covered with safety guards. Therefore, the compressors were screened at a distance of six to 12 inches from the seals. One of the compressor seal systems used an ethylene buffer gas. This seal system was vented to the flare header.

Thirty relief valves were connected directly to the flare header and were therefore not screened. One pressure relief valve which vented to a process drain was screened.

3. SAMPLING TIME

Screening of the vinyl acetate process unit was accomplished by a two-person team in seven working days from March 4 to March 12, 1980.

About 3,330 sources were identified, of which 2,780 were actually screened. Including time spent for instrument calibration, the screening effort required 42 hours (84 person-hours). Sources were screened at an average rate of 0.9 minute per source for a two-person team (or about 1.8 person-minutes per source).

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT=5

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	69	0	3	4.35	2	2.90
	LIGHT LIQUID	117	0	1	0.85	0	0.00
	HEAVY LIQUID	5	0	0	0.00	0	0.00
PROCESS DRAINS	GAS	63	24	0	0.00	0	0.00
	LIGHT LIQUID	1	0	0	0.00	0	0.00
	HEAVY LIQUID	3	0	0	0.00	0	0.00
OPEN ENDED LINES	GAS	108	34	9	8.33	7	6.48
	LIGHT LIQUID	272	33	11	4.04	8	2.94
	HEAVY LIQUID	14	1	2	14.29	2	14.29
AGITATOR SEALS	GAS	1	2	0	0.00	0	0.00
RELIEF VALVES	GAS	1	23	1	100.00	0	0.00
	LIGHT LIQUID	0	7	0	0.00	0	0.00
VALVES	GAS	620	252	37	5.97	23	3.71
	LIGHT LIQUID	1400	170	22	1.57	7	0.50
	HEAVY LIQUID	57	4	0	0.00	0	0.00
PUMPS	LIGHT LIQUID	45	4	1	2.22	1	2.22
COMPRESSORS	GAS	4	0	0	0.00	0	0.00

APPENDIX L

VINYL CHLORIDE MONOMER AND ETHYLENE DICHLORIDE

PROCESS DESCRIPTION, PLANT A - PROCESS UNITS 28 AND 29

Plant A uses the balance process to produce vinyl chloride monomer (VCM). The process is so named because all of the intermediate HCl and ethylene dichloride (EDC) produced is recycled in the process. The net process reaction is:

$$4C_2H_4 + 2Cl_2 + O_2 \rightarrow 4C_2H_3Cl + 2H_2O$$

Three principal reaction steps are involved:

1. Catalytic chlorination of ethylene.
2. Oxychlorination of ethylene.
3. EDC cracking (pyrolysis).

The impurities and moisture introduced in conjunction with reaction Steps 1 and 2 are removed in a purification step, which precedes Step 3. Product VCM is separated from EDC and HCl in a VCM column. Figure 1 illustrates the basic process flow.

Chlorination

Chlorination of ethylene produces crude EDC by the following catalytic reaction:

$$\underset{\text{(ethylene)}}{C_2H_4} + \underset{\text{(chlorine)}}{Cl_2} \rightarrow \underset{\text{(ethylene dichloride)}}{C_2H_2Cl_2}$$

This reaction occurs in the Low Temperature Chlorination-Ethylene Dichloride (LTC-EDC) reactor. Because the reaction is exothermic, care must be taken to maintain temperature control. The liquid EDC is drawn off the reactor and is

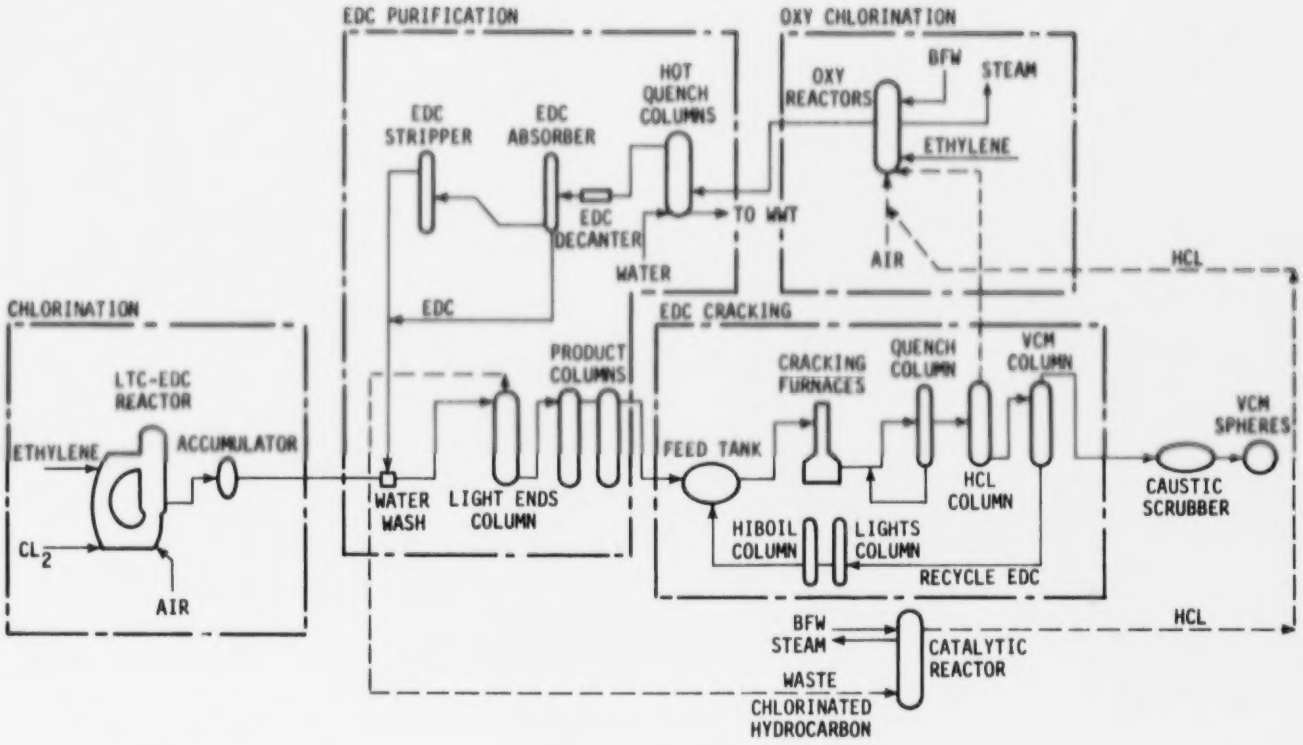

Figure 1. VCM balance process flow diagram.

water scrubbed to remove catalyst and unreacted chlorine before it continues through the EDC purification train.

Oxychlorination

Hydrogen chloride and ethylene are byproducts of VCM production. Additional HCl is produced as a byproduct from a catalytic oxidation reactor that serves another process in the plant. A mixture of HCl, oxygen, and ethlylene under gas catalytic reaction to produce crude EDC as follows:

$$\underset{\text{(ethylene)}}{2C_2H_4} + \underset{\text{(oxygen)}}{O_2} + \underset{\text{(hydrogen chloride)}}{4HCl} \rightarrow \underset{\text{(ethylene dichloride)}}{2C_2H_2Cl_2} + \underset{\text{(water)}}{2H_2O}$$

The oxychlorination reaction is exothermic; proper temperature control must be maintained to ensure stability. The products are quenched and cooled before they enter a decanter, in which the crude EDC and water are separated. This EDC is combined with the crude EDC stream from the chlorination reactor for purification.

The remaining material from the decanter is condensed with cold brine. This stream is then fed into a kerosene absorber, which recovers EDC from the nitrogen and other inert gases remaining from the oxychlorination process. The absorbed EDC enters a stripper, which separates the EDC from the absorbent. The absorbent is recycled, and the recovered EDC enters the crude EDC line for purification. This step completes the oxychlorination cycle.

EDC Purification

Streams of crude EDC from the chlorination and oxychlorination processes are combined into a single stream, which is sent through a column for removal of the light ends. The bottom stream proceeds to the product columns for removal of the high-boiling materials. The EDC is dried in the drying column before it is cracked.

VCM Production

Production of VCM begins by cracking pure EDC into VCM and HCl in thirteen pyrolysis furnaces:

$$\underset{\text{(ethylene dichloride)}}{C_2H_2Cl_2} \xrightarrow{\text{heat}} \underset{\text{(vinyl chloride monomer)}}{C_2H_3Cl} + \underset{\text{(hydrogen chloride)}}{HCl}$$

The product is immediately quenched to force the net reaction to the right and to separate the EDC, VCM, and HCl. The mixed stream from the quench column enters the HCl column, where the HCl is separated from the EDC and VCM for recycle to the oxychlorination process. The HCl column bottoms then pass through the VCM column, which removes the last remaining EDC for recycle. The EDC recycle stream passes through two columns that remove contaminants before the stream is sent to the EDC tank that feeds the cracking process.

The remnants of HCl are removed from the HCl column overhead in a caustic scrubber before the stream proceeds to VCM storage, from which the final product is distributed.

SAMPLING PROBLEMS AND OBSERVATIONS - PLANT A

The most important of several quality control procedures was daily calibration of the hydrocarbon detectors. In addition to the morning calibration recommended by EPA, PEDCo conducted an evening check for calibration drift. The evening check data are included on the calibration checking form and control charts. The technique for calibration of the OVA-108 was established by EPA; the procedure for calibration of the OVA-128 was adapted for use with the dilution probe. The high standard calibration was adjusted with the pinch clamp regulating the airflow through the diluter. The gas select knob was used to adjust the instrument to the low calibration standard without the use of the dilution probe, with the instrument operating in the 100X mode.

The dilution probe carbon pack was changed every other day to ensure proper operation. For calibrating purposes the low and high calibration standards consisted of methane gas at concentrations of 491 ppm and 8450 ppm.

The repeat screening procedure required by EPA was performed on three sources with leaks in different ranges of concentration. Repeat screenings were performed only on the second day, because a shutdown had been scheduled for the end of the week and we wanted to complete the testing in the allotted time.

Screening of the VCM and EDC process units was arranged so as to facilitate data collection and to prevent duplication of screening efforts. First, both screening teams covered the VCM manufacturing and purification plant

(Process Unit 29). One team then screened the EDC manufacturing and purification steps (Process Unit 28) while the other team completed the VCM screening.

The PEDCo team designated process unit codes with the help of the host company. Plant A includes five units; two EDC production units were designated code "28", and three VCM production units were designated code "29." We completed he material coding sheets before entering the plant to facilitate data collection and to eliminate the need for communication while collecting data.

The flame-ionization hydrocarbon instruments used by PEDCo in the survey were produced by Century Systems. Two Organic Vapor Analyzer (OVA) models were used: the OVA-108 and the OVA-128 with a dilution probe. The only difference in the two models is that the OVA-108 has a log-scale readout with a range from 1 ppmv to 1 percent, whereas the OVA-128 has a linear readout with a range from 1 to 1000 ppmv. Use of the dilution probe on the OVA-128 provides capabilities of up to 10,000 ppmv at a 10 to 1 dilution of the sample stream. In this study the OVA-108 was assigned code "1" and the OVA-129, code "2".

The Tygon tubing used on the OVA to prevent probe fouling absorbed hydrocarbons when exposed to strong leaks; this affected the OVA response time considerably. The problem was readily solved by carrying spare tips to replace those that fouled, and eventually replacing the Tygon with Teflon tubing. Another problem was unavoidable aspiration of liquid contaminants, especially around pump and compressor seals. This usually extinguished the hydrogen flame, necessitating additional time to purge and relight the OVA.

An instrument defect in the OVA-128 also caused extinguishing of the hydrogen flame. This defect was apparent when the scale of the instrument was switched, because the recorder unit did not respond accordingly. When the OVA was operated at another plant in the screening study, this defect was found to result from an electrical short in the combustion chamber, and a new thermocouple was installed. A flameout can be detected immediately by repeat screening when a value shows high concentration or by exposing the probe to a hydrocarbon source and observing the response.

Operation of the OVA-128 proved to be awkward in comparison with that of the OVA-108. Access to the multiplier switch was limited because the cover must be left on to prevent damage to the instrument. The use of gloves made

this problem worse and often led to accidential activation of the wrong switch. Evening calibration checks indicated large drifts, attributable to loosening of the pinch clamp on the dilution probe. The pinch clamp should be replaced with a needle valve. Although the OVA-108 was superior for this application because of its simple operating technique, operation was not problem free. Adjustments of the internal electronic calibration were needed regularly.

Time was also a factor. The screening at Plant A was advanced because of a rescheduled plant shutdown. The PEDCo team had to deliver calibration gas and fuel cylinders by car, lacking time to ship them by freight. Also, additional time was needed to complete the screening of the many sources to be checked. All of the five EDC and VCM plants were screened.

The number of flanges screened during the study appears low. The team attempted to screen one of every twenty flanges. The slightly low number of flanges screened could result from inability of the team to see all of the high flanges. Many sources were not screened either because they were too high or too difficult to reach safely from a ladder. Other sources could not be sampled because of dripping liquid, which prevented use of the instrument. Entry of liquid into the probe would foul the OVA.

SAMPLING TIME - PLANT A

Two teams of two persons each screened Process Units 28 and 29 in 176 labor-hours.

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT=28

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	6	0	2	33.33	2	33.33
	LIGHT LIQUID	7	1	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	3	0	2	66.67	1	33.33
OPEN ENDED LINES	GAS	25	2	2	8.00	2	8.00
	LIGHT LIQUID	127	9	22	17.32	13	10.24
RELIEF VALVES	GAS	5	2	0	0.00	0	0.00
	LIGHT LIQUID	2	1	0	0.00	0	0.00
VALVES	GAS	136	32	29	21.32	24	17.65
	LIGHT LIQUID	412	62	16	3.88	8	1.94
PUMPS	LIGHT LIQUID	21	1	6	28.57	3	14.29

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT=29

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	23	0	1	4.35	1	4.35
	LIGHT LIQUID	118	1	1	0.85	1	0.85
OPEN ENDED LINES	GAS	96	19	1	1.04	0	0.00
	LIGHT LIQUID	431	59	31	7.19	14	3.25
AGITATOR SEALS	GAS	0	4	0	0.00	0	0.00
RELIEF VALVES	GAS	1	3	0	0.00	0	0.00
VALVES	GAS	323	97	10	3.10	4	1.24
	LIGHT LIQUID	1580	226	79	5.00	24	1.52
PUMPS	LIGHT LIQUID	47	4	6	12.77	3	6.38

PROCESS DESCRIPTION, PLANT B - PROCESS UNITS 20 AND 21

Plant B also uses the balance process to produce VCM. Principal steps in the process are chlorination, oxychlorination, EDC purification, and VCM production. Figure 2 illustrates the process employed at Plant B.

Chlorination

The chlorination step is similar to that at Plant A, involving the same reaction and equipment. From the reactor the EDC is sent to dry crude EDC storage. The crude EDC is then pumped to a water wash cycle to remove any catalyst that remains in the stream and as much dissolved HCl as possible.

Oxychlorination

The oxychlorination step at Plant B is essentially the same as that at Plant A.

EDC Purification

Streams of crude EDC from both the chlorination and oxychlorination processes are combined into a single stream. The first step in the production of pure EDC is the caustic wash cycle, in which the EDC is washed in a sodium hydroxide (NaOH) solution to remove all HCl residue before it is sent to wet EDC storage. From storage the EDC is sent through a column that removes the light ends for storage and subsequent disposal or sale. The bottom stream from the light ends column proceeds to the heavy ends column, which removes the last remaining impurities. The EDC is sent to storage at a purity of 99.996 percent. At this point the EDC cycle is complete, and production of VCM begins.

VCM Production

Production of VCM begins by cracking pure EDC into VCM and HCl in pyrolysis furnaces, in the same reaction as at Plant A. The product is immediately quenched to prevent the reverse reaction from occuring. Rapid quenching is also necessary to prevent substantial product loss to heavy ends and tars. Any remaining EDC is removed with the tars and sent through tar stills and an EDC recovery column for recycle with the crude EDC stream.

The VCM-HCl product enters the VCM purification train from the quench column. The HCl is first separated from the VCM in an HCl column and is ready

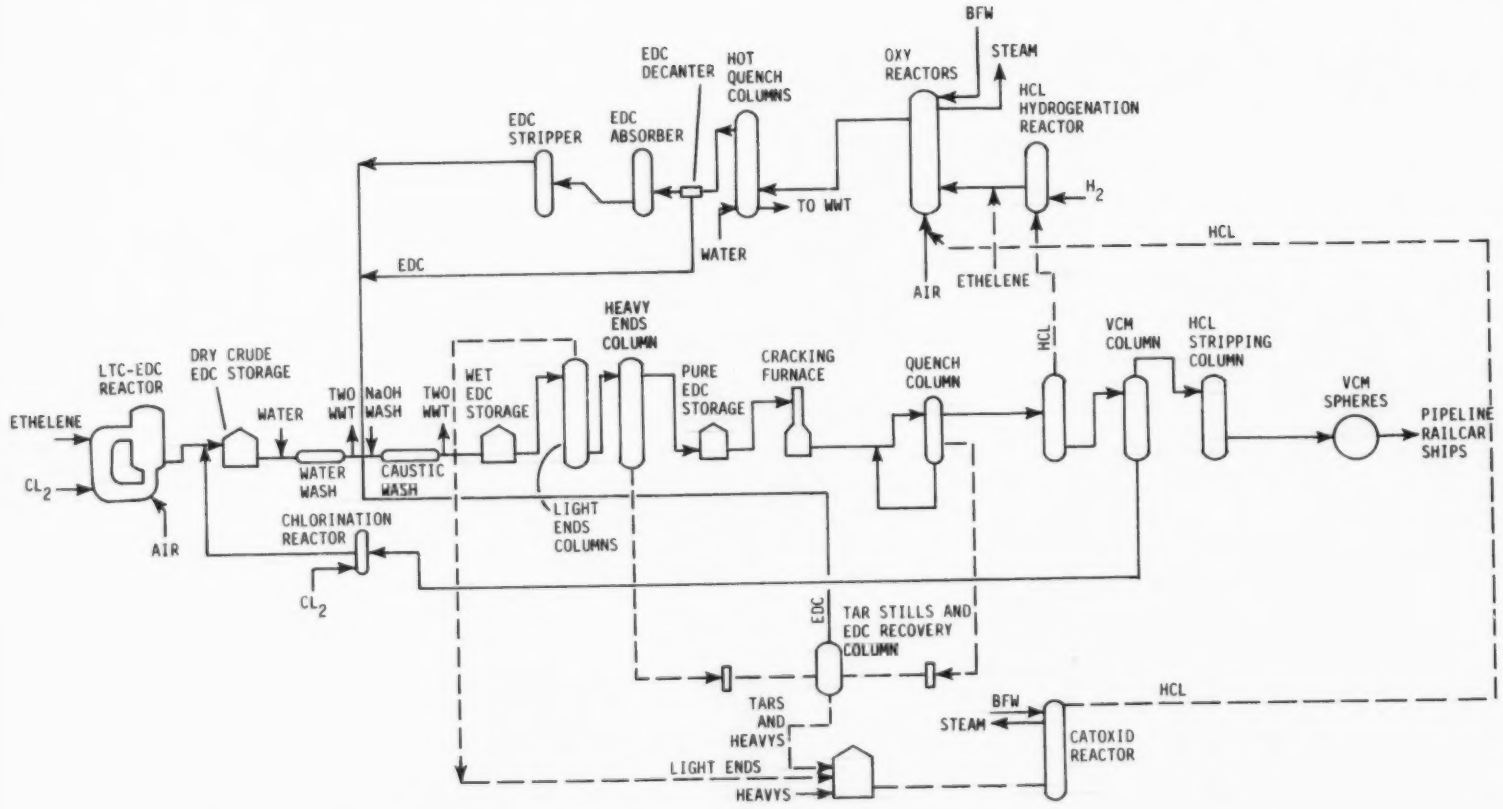

Figure 2. VCM process employed at Plant B.

for use in the oxychlorination process. The stream then passes through the VCM column, which removes the last remaining EDC for recycle. This EDC stream contains chloroprene, benzene, trichloroethane and other impurities that collected in the EDC purification train if left untreated. Chlorinating the EDC recycle stream removes these impurities and prevents contamination.

The final traces of HCl are removed from the main stream in an HCl stripping column. The final VCM product is stored, to be distributed by pipeline, rail, or ship.

SAMPLING PROBLEMS AND OBSERVATIONS - PLANT B

Quality control procedures were the same as at Plant A, including the daily calibration and evening check of the hydrocarbon detectors. The equipment was operated in the same mode as at Plant A, with the OVA-128 adapted for use with the dilution probe. Again, the low and high calibration standards were methane gas at concentrations of 491 ppm and 8450 ppm.

Repeat screening was done by the procedure outlined in the EPA manual when practical. One difficulty was locating three sources for repeat screening within the concentration ranges recommended by the manual. Because the facility was relatively leak-free, we did not locate three repeatable sources until the second day, and the repeating source data were thus incomplete until the third day.

Again, the VCM and EDC process units were screened in sections by two teams. The VCM unit (Process Unit 21) included the pyrolysis furnaces/ quench area and VCM purification. The EDC unit (Process Unit 20) included EDC process area and oxychlorination. One team screened the EDC process area while the other team screened the pyrolysis furnaces/ quench areas, VCM purification, and oxychlorination. Both teams screened approximately the same number of emission sources.

Plant B conducts a very active leak control program. In a VCM plant, the VOC emissions represent a product loss, and recovery can lead to financial savings as well reducing emissions. At the request of plant officials, all points of leakage were marked with green tape so that the leaks could be repaired and the line sizes were recorded in the last column of the data sheets. As part of the leak-control program, most of the flanges had been taped with duct tape with only a pinhole open for screening. We were asked to

remove the tape from flanges that were found to leak, and attempt to locate and mark the site of the leak. Nonleaking sources were marked with a red "X" to prevent duplication.

Another instance of special handling was the screening of sample stations. Many of these stations had a dozen or so emission sources, including two open-ended lines to which sample bottles are attached. Under normal operating conditions there is no product material in the lines, because samples are taken only two or three times daily. We detected substantial amounts of hydrocarbons in the open ended lines, but this is attributed to residue from the sampling operation rather than to leakage. As a means of streamlining the procedure, we decided to screen only the main valves with which the sampling stations were connected to the main process lines.

Coding of the instruments was as at Plant A: "1" for the OVA-108 and "2" for the OVA-128. Process unit codes, determined made with the help of the host company, were EDC production, designated code "20", and VCM production, designated code "21." The material coding sheet was completed before we entered the plant on the basis of piping and instrumentation diagrams provided by plant personnel.

Overall, the screening program went relatively smoothly in spite of several problems that slowed the progress of the screening teams. These problems arose from weather hazards, operation of delicate instruments under field conditions, and dependence on the host company. We tried to solve these problems expeditiously without sacrificing data reliability.

Weather conditions presented unavoidable hazards to team operations. High humidity and an atmospheric temperature inversion on one day kept emissions at ground level, and background concentrations as high as 1400 ppm caused discomfort to the screening teams. A rain shower stopped the screening temporarily. Experimental burning of chlorinated hydrocarbons released HCl into the atmosphere and on two occasions forced one team to descend the column they were screening. Further attempts to screen this column were abandoned until another day. High winds and cold weather prohibited safe screening of sources ordinarily accessible from column ladders. Because of near-freezing temperatures, we wore gloves and bulky clothing and had warmup periods before starting the screening operations.

The instrumentation problems were the same as at Plant A, involving the Tygon tubing used on the OVA and also the awkwardness of operating the OVA-128, especially when wearing gloves. Again the OVA-108 proved to be superior for this application, despite the variation in internal electronic calibration and the need for regular adjustments.

Despite these minor problems the task went relatively smoothly. Officials of Plant B recognize the value of such a survey and expect to benefit from the data collected. Cooperation of plant personnel in the collection of data was invaluable.

The apparent low number of flanges screened (the goal was 5 percent of all flanges) is attributable to the inaccessibility of many of the flanges.

SAMPLING TIME - PLANT B

Two teams screened Process Units 20 and 21 in 200 labor-hours.

PUMP SEALS - PLANT B

Plant B has several dual pump seal applications. Tandem seals are used on pumps in VCM service, with lube oil as the barrier fluid. Back-to-back seals are used for pumps in tar and hydrogen chloride service; hexane is the barrier fluid for this application.

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT=20

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	10	0	0	0.00	0	0.00
	LIGHT LIQUID	40	1	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	2	0	2	100.00	1	50.00
OPEN ENDED LINES	GAS	30	13	2	6.67	0	0.00
	LIGHT LIQUID	213	17	16	7.51	5	2.35
AGITATOR SEALS	GAS	2	0	0	0.00	0	0.00
	LIGHT LIQUID	1	0	0	0.00	0	0.00
RELIEF VALVES	GAS	7	16	0	0.00	0	0.00
	LIGHT LIQUID	7	0	0	0.00	0	0.00
VALVES	GAS	276	114	12	4.35	6	2.17
	LIGHT LIQUID	798	118	14	1.75	4	0.50
PUMPS	LIGHT LIQUID	44	1	5	11.36	4	9.09

4. DATA SUMMARY

SUMMARY STATISTICS
UNIT=21

SOURCE	SERVICE	NUMBER SCREENED	NUMBER NOT SCREENED	NUMBER >=1000	PERCENT >=1000	NUMBER >=10000	PERCENT >=10000
FLANGES	GAS	2	1	0	0.00	0	0.00
	LIGHT LIQUID	45	1	0	0.00	0	0.00
PROCESS DRAINS	LIGHT LIQUID	0	1	0	0.00	0	0.00
OPEN ENDED LINES	GAS	4	0	0	0.00	0	0.00
	LIGHT LIQUID	42	2	3	7.14	2	4.76
RELIEF VALVES	GAS	6	2	0	0.00	0	0.00
	LIGHT LIQUID	10	0	0	0.00	0	0.00
VALVES	GAS	79	14	2	2.53	0	0.00
	LIGHT LIQUID	670	41	5	0.75	0	0.00
PUMPS	LIGHT LIQUID	10	0	0	0.00	0	0.00
OTHER[1]	LIGHT LIQUID	0	1	0	0.00	0	0.00

[1]Expansion joint.